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OXIDATION OF ELEMENTAL SULPHUR FERTILIZERS IN AGRICULTURAL SOILS OF NORTHERN ALBERTA AND SASKATCHEWAN

by

ELSTON D. SOLBERG

(C)

A THESIS

SUBMITTED TO THE FACULTY OF GRADUATE STUDIES AND RESEARCH

IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE

OF MASTER OF SCIENCE

IN

SOIL FERTILITY

DEPARTMENT OF SOIL SCIENCE

EDMONTON, ALBERTA

SPRING 1986

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The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies and Research, for acceptance, a thesis entitled OXIDATION OF ELEMENTAL SULPHUR FERTILIZERS IN AGRICULTURAL SOILS OF NORTHERN ALBERTA AND SASKATCHEWAN submitted by ELSTON D. SOLBERG in partial fulfilment of the requirements for the degree of MASTER OF SCIENCE.

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ABSTRACT

The recovery of applied elemental sulphur as sulphate, with time, was determined in seven laboratory incubation experiments and in six field experiments. The field experiments involved five soils at four locations. Recovery from spring 1980 incorporated Fine S, in two field experiments, was 20-68% two to three months after application and 33-95% in the fall. In another experiment, on four soils in 1981, the recovery of Fine S was 24-58% two to three months after application. The spring (May) recovery of Fine S applied late in the fall, to three soils, was low (7-11%) while the fall recovery was 14-71%. In another experiment, recovery of Fine S approximately one year after application was 27-69%. Bentonitic S generally gave low recoveries relative to Fine S.

Urea S generally gave rates of recovery similar to Fine S in field and incubation experiments but method of application and "cultivation" were more important. In the field, cultivation occasionally increased the rate of oxidation but often did not.

Two cropped field experiments, one with barley and one with bromegrass demonstrated the delay in availability to crops of Fine S and Urea S. A significant residual quality was also observed. With application of larger amounts of Urea S (providing S at 70 kg ha^{-1}) and especially with early application, elemental sulphur was shown to supply sufficient sulphur to the crop the year of application and also to a crop grown the following year.

In laboratory incubations banding and placement (at one distinct point) of elemental sulphur reduced the recovery as sulphate, but a thorough mixing ("cultivation") of the soil increased sulphur recovery. A less intense method of soil mixing ("cultivation") had little effect on the recovery of banded elemental sulphur. Maximum rates of oxidation were observed near 90% field capacity and significant quantities of elemental sulphur were oxidized at all moisture levels investigated. The rate of oxidation at 36°C was determined to be apporximately 4.8 to 6.0 times greater than at 6°C. Restoration of the oxidative capacity of a Breton soil was observed to be progressively slowed as temperature decreased.

Soil samples were taken from four field experimental locations where elemental sulphur had been applied one or two years previously. Samples that had never received elemental sulphur were also obtained from adjacent controls and from "remote" soils. In laboratory incubations, oxidation rates were slowest with "remote" soils and most rapid with soils previously treated with elemental sulphur. The adjacent controls were intermediate.

All of the soils evaluated in this study had significant oxidative capacities under various field and laboratory incubation conditions.

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I. INTRODUCTION

Sulphur deficiencies in legumes, grown on Luvisolic soils low in plant available sulphate-sulphur, were found by Newton (1936) some 50 years ago. These deficiencies of sulphur for legumes soon became moderately common on Luvisolic soils in Central Alberta but the deficiency did not seem to affect non-leguminous crops. With hindsight, it can be deduced that non-legumes would have displayed the symptoms of sulphur deficiency had they been receiving applications of nitrogen (Robertson 1979).

In the Peace River region, during the late 1960's, sulphur fertilization was observed to give large yield increases of cereal grain and canola (Brassica campestris) crops grown on low sulphur soils provided sufficient nitrogen was also added (Nyborg 1967, Nyborg et al. 1974). Reports ensued of sulphur deficiencies in cereal and canola crops on low sulphate-sulphur Luvisolic and Chernozemic soils across the Prairies.

In Alberta it is estimated that 1.6 million ha are presently sulphur deficient for cereals while 3.0 million ha are sulphur deficient for canola and legumes (Laverty 1985). Further, approximately 4 million ha of cultivated soils in the Prairie Provinces are now sulphur deficient with an estimated additional 8 million ha being potentially sulphur deficient (Bettany et al. 1983).

Sulphur fertilizers commonly used in the Prairie Provinces contain soluble sulphate rather than elemental sulphur. Sulphate

fertilizers are effective in rapidly overcoming crop sulphur deficiencies (Ukrainetz 1982, Janzen and Bettany 1984b). The main sulphate sources are ammonium sulphate, blends of urea and ammonium sulphate, or ammonium phosphate-sulphate while materials such as $CaSO_4$, Na_2SO_4 and K_2SO_4, although effective, have seldom been utilized.

For cereal grains and canola, there has been a lack of understanding among farmers, fertilizer agents and occasionally agronomists regarding the amount of sulphur fertilizer required in relation to the amount of applied fertilizer nitrogen for sulphur deficient soils. Generally the amount of fertilizer sulphur applied should be one-tenth of the amount of fertilizer nitrogen applied. For cereals, the applied N:S ratio can often be as wide as 15:1 (Stewart et al. 1966) while a more dependable ratio for general canola use would be 7:1 (Janzen and Bettany 1984a). Application of still narrower N:S fertilizer ratios (<7:1) is acceptable providing sufficient nitrogen is applied to overcome any nitrogen deficiency. It was against this involved setting that elemental sulphur fertilizers appeared on the Prairie Provinces in the late 1970's to further compound the sulphur situation.

For fertilizer manufacturers and farmers, elemental sulphur has the advantages of ready supply, lower production costs, and, because of its high analysis, lower transportation costs. However, elemental sulphur requires biological oxidation to plant available sulphate before it can be utilized by crops. The factors affecting oxidation have been studied throughout the world for the past 50

years but only very recently in the Prairie Provinces. The recent interest in elemental sulphur arose when a product touted as being rapidly oxidizable was introduced and subsequently found to be of little benefit when spring applied to canola on sulphur deficient soils. From this it was evident that results from other areas could not be applied to the Prairie Provinces. Results from research conducted in the Prairie Provinces have been, not unexpectedly, inconsistent for elemental sulphur forms and also occasionally variable from researcher to researcher.

The present study was undertaken with the following objectives:

1) to determine the effect of environmental factors on the rate of oxidation of elemental sulphur.
2) to evaluate the influence of previous applications of elemental sulphur on the oxidation rate of subsequent applications.
3) to investigate the effect of various methods of application on the oxidation of different elemental sulphur sources.
4) to evaluate the effect of cultivation on elemental sulphur oxidation.
5) to investigate the short-term and residual plant availability of elemental sulphur.

2. LITERATURE REVIEW

The purpose of this literature review is to investigate the factors affecting the oxidation of elemental sulphur fertilizers to plant available sulphur and to examine how these factors affect the elemental sulphur's ability to augment the soil reserves of plant available sulphur so that crop requirements can be fulfilled.

2.1 SOIL SULPHUR

The soil is the primary supplier of plant available sulphate sulphur to growing crops. In non-saline soils more than 95% of the soil sulphur is held in organic forms (Tabatabai and Bremner 1972, Bettany et al. 1973). The organic matter of agricultural soils contains from 0.005 to 0.05% sulphur and from 0.04 to 0.4% nitrogen (Nyborg 1978). Organic sulphur has been divided into three fractions summarized by Williams (1975) as follows: hydriodic acid-reducible sulphur (ester sulphate), carbon-bonded sulphur and Raney-nickel-reducible sulphur (subfraction of carbon-bonded sulphur). Hydriodic acid-reducible sulphur ranges from 30 to 70% of the total organic sulphur while carbon-bonded sulphur on average comprises about 50%. The Raney-nickel-reducible subfraction of carbon-bonded sulphur varies from soil to soil but averages 25 to 30% of the total organic sulphur.

Inorganic sulphur includes reduced compounds of sulphide, polysuiphide, sulphate, thiosulphate and elemental sulphur but is

principally comprised of soluble and adsorbed sulphate in soils that are not water logged (Williams 1975). Bettany et al. (1973) found that extractable sulphur ranged from 0.5 to 13.5 ug g^{-1}, which represented only 0.5 to 3.6% of the total sulphur in some Saskatchewan soils.

Each year about 1 to 3% of the organic sulphur is mineralized to sulphate and a similar amount of sulphate is immobilized by incorporation into the soil organic matter (Nyborg 1978). The process of mineralization and immobilization are primarily microbiologically mediated (Freney et al. 1962, Alexander 1977, Zinder and Brock 1978). The amount of organic sulphur mineralized to plant available sulphate depends mainly upon the N:S ratio of added organic matter and not on the stable N:S ratio of soil organic matter (Freney et al. 1962).

Additions of sulphur from the atmosphere can be sufficient to alleviate potential sulphur deficiencies. Soils and plants near industrialized areas have been shown to absorb emitted sulphur dioxide (Nyborg 1978, Olson 1957) while rain can contribute up to 4 kg S ha^{-1} in Alberta (Nyborg and Walker 1977). Particulate deposition can add significant quantities of sulphur in localized areas (Nyborg 1978, Wainwright 1979, Wainwright and Killham 1982). Further anthropogenic additions of sulphur occur through application of herbicides, insecticides and fungicides and with irrigation water.

Plant available sulphate sulphur can be lost from the rooting zone by immobilization or by leaching. The anionic sulphate ion is mobile and subject to movement down the soil profile with

extensive or intense periods of precipitation. The extent of losses depends upon the concentration of sulphate in solution, the moisture retention capacity of the soil, hydraulic conductivity of subsurface horizons, the volume of percolating water, and the type of plant growth (Rhue and Kamprath 1973).

2.2 CROP REQUIREMENTS

Sulphur deficiency generally occurs when the soil is unable to supply sufficient sulphate to fast-growing crops. If the soil sulphate is not augmented by atmospheric sulphur, then fertilizer sulphur must be added to give optimal yields.

Typical requirements of the above-ground portion of crops commonly grown on the Prairie Provinces range from 20 to 39 kg S ha^{-1} for legumes and canola while 18 to 28 kg S ha^{-1} is required by grasses and cereal grains (Beaton and Fox 1971).

Field studies in the Prairie Provinces indicate that applications of 20 to 30 kg S ha^{-1} (often 10 kg S ha^{-1}) in the sulphate form is sufficient to obtain high crop quality and yield (Nyborg et al. 1974, Ukrainetz et al. 1975). The rates of sulphur (in sulphate form) recommended by the provincial soil test laboratories in Saskatchewan and Alberta for severely sulphur deficient soils is 15 kg ha^{-1} for cereals and 20 to 30 kg ha^{-1} for legumes and canola.

2.3 OXIDATION OF ELEMENTAL SULPHUR

Elemental sulphur can be oxidized in soil through chemical processes but these are usually much slower and therefore of less importance than microbial oxidation (Weir 1975). Studies have shown significant oxidation of elemental sulphur by sterilized soils but these observations were attributed to contamination or inadequate sterilization (Vitolins and Swaby 1969, Wainwright and Killham 1980, Nor and Tabatabai 1977).

Considerable controversy exists about the biochemical pathways of elemental sulphur oxidation and most of what we know comes from studies of Thiobacillus spp. (Grant and Long 1981). Oxidation of elemental sulphur to sulphate is believed to require direct microbial contact (Vogler and Umbreit 1941) and proceeds via the polythionate pathway in which elemental sulphur is oxidized to sulphate through thiosulphate and a series of polythionate intermediates ($^{-}O_3SS_nSO_3^{-}$) where n may be tetrathionate ($S_4O_6^{=}$), trithionate ($S_3O_6^{=}$) and sulphite ($SO_3^{=}$) (Alexander 1977, Nor and Tabatabai 1977).

Biological oxidation is affected by:

i) environmental conditions in the soil;

ii) microfloral population of the soil; and

iii) characteristics of the elemental sulphur source and cultural methods during and after application.

2.3.1 Soil Microfloral Population

A number of organisms, including heterotrophs, photoautotrophs and chemoautotrophs, are capable of oxidizing elemental sulphur. The autotrophic Thiobaccillus spp. are usually considered to be the most important group of oxidizers and have been studied extensively (Vishniac and Santer 1957, Starkey 1966, Vishniac 1974) while the heterotrophic fungi, actinomycetes and bacteria involved in sulphur oxidation have been largely ignored.

Soils receiving large amounts of elemental sulphur have been shown to have increased numbers of Thiobacillus and an increased oxidative capacity (Moser and Olson 1953, Bertrand 1973) as compared to untreated soils. Vitolins and Swaby (1969) could show no effect of agronomic rates of application of elemental sulphur fertilizer on subsequent oxidative activity. The numbers of sulphur oxidizing microorganisms (especially autotrophs) in agricultural soils are low (Vitolins and Swaby 1969, Germida et al. 1985) and there would not seem to be any correlation of soil oxidizing capacity and population composition (Kuenen 1975, Wainwright 1978).

Laboratory soil inoculation with suspensions of previously elemental sulphur enriched soil or cultures of sulphur oxidizing organisms (heterotrophs, autotrophs and mixtures of both) have often given increased rates of sulphur oxidation (Kittams and Attoe 1965, Li and Caldwell 1966, Rupela and Tauro 1973, McCready and Krouse 1982). Field inoculation generally has not had similar success (Weir 1975).

The importance of heterotrophic sulphur oxidizing microorganisms should not be understated. Vitolins and Swaby (1969)

suggested that heterotrophic sulphur oxidizers may be the primary oxidizers of reduced sulphur in soils until the pH is sufficiently lowered for secondary oxidation by Thiobacillus spp. Pepper and Miller (1977) isolated heterotrophic bacteria that oxidized appreciable amounts of elemental sulphur in liquid medium and autoclaved soil. Following this, several other heterotrophic microorganisms, including actinomycetes, bacteria and fungi, have been shown to oxidize elemental sulphur (Wainwright and Killham 1980). In Saskatchewan, where fine elemental sulphur fertilizers have been rapidly oxidized (Ukrainetz 1982, Janzen and Stewart 1982), no autotrophic sulphur oxidizing Thiobacillus could be isolated from the soils examined (Germida et al. 1985). Further, stock cultures of associative and symbiotic nitrogen-fixing bacteria (i.e., Azospirillum and Rhizobium spp.) were observed to slowly oxidize elemental sulphur at rates significantly lower than Thiobacillus.

2.3.2 Environmental Conditions in the Soil

Optimal oxidation of elemental sulphur occurs when soil moisture is at or slightly above field capacity (Moser and Olson 1953, Kittams and Attoe 1965). Moser and Olson (1953) pointed out that oxidation occurred at all moisture levels ranging from less than air dry to saturation. At moisture extremes, limitations of oxygen (excessively wet) and metabolic water (excessively dry) cause the oxidation rate to decrease dramatically but oxidation still occurs. Janzen (1984) found a parabolic relationship between soil moisture and sulphur oxidation similar to that of Moser and Olson (1953).

Sulphur oxidation occurs over a wide range of temperatures with the optimum rates occurring between 27 and 35°C. Li and Caldwell (1966) found rates of oxidation four times greater at 30°C than at 23°C. Similarily, Nor and Tabatabai (1977), after 74 days of incubation, observed that rates of oxidation were 2.3 to 6.1 times faster at 30°C than at 5°C and 1.7 to 3.7 times faster at 30°C than at 15°C. Janzen (1984) found an exponential relationship between temperature and sulphur oxidation whereby each 5°C increase in temperature gave a doubling of oxidation rate.

Rehm and Caldwell (1969) observed that, while not statistically significant, coarse textured soils generally oxidized elemental sulphur more quickly than fine textured soils. Oxidation rates have generally been observed to be more rapid in neutral to slightly alkaline soils than in acid soils (Kittams and Attoe 1965, Nor and Tabatabai 1977).

The addition of plant residues or carbon substrates have variably affected the rate of sulphur oxidation (Li and Caldwell 1966, Vitolins and Swaby 1969, Weir 1975). Pepper and Miller (1978) found that the addition of glucose to pure cultures stimulated heterotrophic sulphur oxidation but inhibited autotrophic oxidation. Stewart (1966) added straw of differing sulphur content and found that straw with less than 0.15% S decreased greenhouse wheat yields when fertilizer S was not applied. This implies that the addition of plant residues affect the overall mineralization-immobilization process rather than the rate of elemental sulphur oxidation.

Tisdale et al. (1985) state that large differences observed in the initial rates of sulphur oxidation by soils are unrelated to soil type or various properties including texture, organic matter, field capacity, pH, initial sulphate sulphur or degree of sulphur deficiency. Janzen (1984) selected 40 diverse Saskatchewan soils for incubation and found several soil characteristics correlated to sulphur oxidation. Oxidation rates were positively correlated to pH, air filled porosity, organic carbon content, extractable P and extractable sulphate while inversely related to clay content and moisture content at 0.3 atmospheres tension.

2.3.3 Elemental Sulphur Characteristics and Cultural Methods

The rate of oxidation of elemental sulphur increases as the particle size decreases (Li and Caldwell 1966, Jones and Ruckman 1969, Kemnec et al. 1981). The rate of oxidation of elemental sulphur is linearly related to the specific surface area of the added sulphur material (Fox et al. 1964, Koehler and Roberts 1983).

To maximize oxidation of elemental sulphur it is necessary to thoroughly mix the sulphur into the soil. This maximizes the sulphur particle-soil contact thereby increasing exposure to microbial oxidizers. Banding or placement of elemental sulphur can greatly reduce rates of oxidation through decreased surface area exposure (Nyborg 1980, Ukrainetz 1982). Sulphur materials or methods of application that limit the dispersion and contact of elemental sulphur with the soil will decrease the potential oxidation of the added sulphur. Bentonitic sulphur products have been rather

inadequate short-term suppliers of sulphate sulphur (Nyborg et al. 1980, Noellemeyer et al. 1981, Ukrainetz 1982). Similarily, Urea-S has given slow release of sulphate but a second "cultivation" increased the rate of oxidation (Nyborg et al. 1980, Janzen 1986).

2.3.4 Evaluation of Oxidation Rates in the Literature

Comparison of oxidation rates found in the literature is difficult as researchers have seldom standardized incubation conditions or the particle size of the elemental sulphur used for oxidation. The ability of a soil to oxidize elemental sulphur has been commonly evaluated, in the laboratory or in the field, by incubating soil with elemental sulphur for a predetermined period of time. Sulphate production was then considered to be the proportion of the elemental sulphur oxidized after basal levels were subtracted. However, the measurement of sulphate to estimate elemental sulphur oxidation can be confounded by the mineralization-immobilization processes of soils (Li and Caldwell 1966, Nyborg et al. 1980). This is especially true for long-term laboratory incubations and field experiments.

Evaluation of the oxidative capacity of soils in North America (Kittams and Attoe 1965, Attoe and Olson 1966, Nyborg et al. 1980, Janzen et al. 1982) and Australia (Vitolins and Swaby 1969) have given a wide range of values. Often these differences in oxidation rate have been attributed to differences in the activity and size of the microbial oxidizing population.

Because elemental sulphur is biologically oxidized and is highly dependent upon the elemental sulphur source and method of application (Janzen 1984), comparison of results from different areas and researchers is difficult since incubation and field conditions vary. Further, comparison of field trials from year to year (area to area) is valuable only in a general way.

2.3.5 Summary

The soil is the primary supplier of sulphur to growing crops. When the supply is insufficient for optimal crop growth, sulphur fertilizer is needed. Traditionally, sulphate fertilizer sources have been effective suppliers of plant available sulphur. The recent Prairie Provinces' introduction of fertilizers requiring biological oxidation from elemental sulphur to sulphate have clouded the sulphur deficiency picture. Therefore, further information is required regarding the factors governing the availability of elemental sulphur fertilizers for crop growth.

3. MATERIALS AND METHODS

3.1 SOILS

Laboratory incubation experiments were conducted with surface soils (0-15 cm) taken near field plots at Breton, Ellerslie and Glendon (Alberta); and Canwood (Saskatchewan). Descriptions of the incubation experiment and field plot soils are in Table 1 and Table 2, respectively.

3.2 SULPHUR SOURCES

Several sulphur sources were evaluated in laboratory and field experiments. Some of the materials were experimental while others were commonly available. A description of the evaluated sulphur sources is given in Table 3.

Bentonite S consists of "degradable" granules comprised of 10% bentonite and 90% elemental sulphur. When wetted, the bentonite expands thereby fracturing and dispersing the elemental sulphur into "fine" particles. The three bentonitic S sources used in this study behave similarily in the soil.

Urea S consists of urea pellets impregnated with fine elemental sulphur particles which are released when urea dissolves. Urea S (A) and (B) were experimental formulations while Urea S (C) is commercial.

Flowable S consists of very fine particles (1-2 um) of elemental S in a creamy liquid suspension containing 0.72 kg S L^{-1}. This material has been used in the United States for some time and is new to the Prairie Provinces.

Table 1. Description of soils used in the incubation experiments (0-15 cm).

Location	Soil Great Group	Texture	Soil Water Content (%) Field Capacity	Air Dry	Total N	Mineral N	Total S	SO_4-S	pH 1:2.5
					(- - - - - - - - ug g^{-1} - - - - - - - -)				
Breton	Gray Luvisol	SiL	22.1	1.8	1438	22.4	129	2.5	6.0
Canwood	Gray Luvisol	L	27.3	2.0	1688	18.6	177	2.9	6.5
Canwood Brome	Dark Gray Chernozem	SL	28.6	1.4	3049	13.4	368	2.2	6.6
Ellerslie	Black Chernozem	SiCL	40.0	6.0	3834	66.9	562	7.6	5.8
Glendon	Dark Gray Chernozem	SL	25.0	2.3	2941	81.7	330	13.9	6.7

Table 2. Description of soils at field experiment sites.

Location	Soil Great Group	Depth (cm)	Texture	Bulk Density (Mg m^{-3})	Total N (- - - - - - - ug g^{-1}	Mineral N	Total S - - - - - - -	SO_4-S -)	pH 1:2.5
Breton	Gray Luvisol	0-15	SiL	1.31	1190	26.9	110	2.9	6.2
		15-30		1.42	515	14.8	49	3.0	6.0
		30-60		1.68	349	15.9	37	2.6	6.1
		60-90		1.65	284	3.6	42	2.6	6.0
Canwood	Gray Luvisol	0-15	L	1.29	1720	15.6	163	2.8	6.4
		15-30		1.43	440	3.9	36	2.4	6.8
		30-60		1.50	233	2.3	36	1.9	7.9
		60-90		1.58	74	1.0	6	1.4	8.7
Canwood Brome	Dark Gray Chernozem	0-15	SL	0.80	1996	13.5	390	1.8	6.7
		15-30		1.36	1215	7.1	170	2.4	6.9
Ellerslie	Black Chernozem	0-15	SiCL	0.96	3945	53.7	518	4.0	6.1
		15-30		1.06	3132	41.6	384	2.6	7.0
		30-60		1.22	769	3.4	115	2.4	7.6
		60-90		1.40	530	2.6	103	2.0	7.7
Glendon	Dark Gray Chernozem	0-15	SL	1.26	2802	42.5	326	3.1	6.6
		15-30		1.40	589	6.1	41	1.3	7.1
		30-60		1.50	479	9.6	34	0.9	6.6
		60-90		1.53	340	7.5	32	0.6	7.1

Table 3. Description of sulphur sources evaluated (continued).

Sulphur Source	Characteristics	% N	% S	Diameter Range of S Particle	% by Weight in Diameter Range
Bentonitic S (A)	10% bentonite; granules 2-4 mm diameter; 1979 product	0.0	90	Not done	Not done
Bentonitic S (B)	10% bentonite; dome shaped 2-4 mm long, 1-3 mm wide; 1985 product	0.0	90[2]	>2,000 um	0
				2,000-1,180 um	17
				1,180-600 um	36
				600-250 um	20
				250-150 um	13
				150-75 um	11
				75-45 um	3
				<45 um	1
Bentonitic S (C)	10% bentonite; granules 2-4 mm diameter; 1985 product	0.0	90[2]	>2,000 um	25
				2,000-1,180 um	15
				1,180-600 um	15
				600-250 um	16
				250-150 um	13
				150-75 um	12
				75-45 um	4
				<45 um	1

1 See Appendix V for details of fractionation.
2 See Appendix VI for details of fractionation.

Table 3. Description of sulphur sources evaluated.

Sulphur Source	Characteristics	% N	% S	Diameter Range of S Particle	% by Weight in Diameter Range
Sodium sulphate	Reagent grade	0.0	22.5	Not done	Not done
Urea S (A)	Urea pellets; 1-3 mm diameter; impregnated with fine elemental S	39.6	10.4	25-50 um <25 um	50 50
Urea S (B)	Urea pellets; 1-3 mm diameter; impregnated with fine elemental S	35.7	22.4	25-50 um <25 um	50 50
Urea S (C)	Urea pellets; 1-3 mm diameter; impregnated with fine elemental S	36.0	20.0	25-50 um <25 um	50 50
Flowable S	Elemental S in suspension	0.0	52	1-2 um	100
Fine sublimed S	Reagent grade	0.0	99.5[1]	>250 um 250-150 um 150- 75 um 75- 53 um 53- 45 um < 45 um	0 22.9 58.2 7.7 7.2 4.2

(continued)

Reagent grade fine sublimed elemental sulphur (referred to as Fine S) was commonly used as the experimental elemental sulphur standard. The majority of particles had a diameter of 75 to 150 um but significant quantities of other particle size fractions were present (Table 3).

Excluding flowable sulphur, the elemental sulphur sources all tended to consist of flat, irregularly shaped particles of varying diameters.

3.3 LABORATORY INCUBATION EXPERIMENTS

Seven laboratory incubation experiments were conducted to investigate several of the factors that influence oxidation of elemental sulphur in soils. Many details of each incubation differ but there are some aspects of all the incubation experiments that are similar or identical.

Cylindrical 1000 ml plastic containers (11.0 cm diameter and 13.5 cm high) and cylindrical 500 ml plastic containers (11.0 cm diameter and 7.5 cm high) were used as the incubation containers. To maintain soil water content (and to reduce evaporation), while keeping the incubating soil aerated, plastic lids with a single central hole of 5-6 mm were used on containers. Deionized water was used to moisten the soil samples in containers to their experimentally required weight. Weights of containers plus moist soil were made weekly and the initial weight was maintained through additions of deionized water. All incubation experiments were conducted in darkened incubation chambers where temperatures were maintained with 1°C above or below the target temperature.

Elemental sulphur was applied to the soils when dry and the application methods included:

i) Mixed: even surface application followed by a thorough stirring of the elemental sulphur into the top 7 to 8 cm of the soil.

ii) Placed: application of all of the elemental sulphur to one distinct point in the center of the container at a soil depth of 3 to 4 cm.

iii) Banded: application of all the elemental sulphur in one narrow band in the center of the container at a soil depth of 3 to 4 cm.

iv) Surface: an even application of the elemental sulphur over the soil surface.

The term "cultivation" refers to the mixing of the incubating soil seven or eight days after initiation of incubation. Two methods of "cultivation" were used. One method involved a vigorous clockwise stirring of moist soil for ten rotations to a depth of 8 to 9 cm with a heavy spatula. A second method involved the complete removal of the soil sample from the incubation container onto a plastic sheet where a complete "cultivation" was performed.

At designated sampling dates, predetermined sample containers were removed from the incubating chambers so that the soil could be spread and air dried. Each sample was then ground to pass through a 2 mm sieve in preparation for sulphate analysis.

3.3.1 Oxidation of Elemental Sulphur as Affected by Source, Method of Application and Cultivation

Three incubation experiments were conducted to investigate the effects of cultivation and various methods of application on the rate of oxidation of several elemental sulphur sources.

3.3.1.1 Experiment One

This experiment used the Canwood and Ellerslie soils described in Table 1. Samples of 500 g were weighed into 500 ml plastic containers and treated with one of the following nine treatments with sulphur applied at 25 ug g^{-1}:

i) Control: no sulphur added.
ii) Fine S: mixed.
iii) Fine S: placed.
iv) Sodium sulphate: mixed.
v) Bentonitic S (A): placed.
vi) Bentonitic S (A): mixed.
vii) Urea S (A): mixed.
viii) Urea S (A): mixed and then cultivated.
ix) Urea S (A): placed.

The soils were wetted to 90% field capacity and the containers were incubated in the dark at a constant 26°C for 3 and 12 weeks. After seven days of incubation, Urea S treatment (viii) was "cultivated" by mixing the soil on a plastic sheet.

Each treatment was repeated four times for each soil allowing for removal of duplicate samples at each sampling date.

After removal, the samples were spread and air dried at room temperature.

3.3.1.2 Experiment Two

This experiment utilized the Breton and Ellerslie soils described in Table 1. Samples of Breton (930 g) and Ellerslie (750 g) were weighed into 1000 ml plastic containers and treated with one of the following eight treatments with sulphur applied at 100 ug g^{-1}:

i) Control: no sulphur added.

ii) Sodium Sulphate: mixed.

iii) Bentonitic S (C): mixed.

iv) Bentonitic S (C): mixed and then cultivated.

v) Bentonitic S (C): ground to give particles that were less than 150 um in diameter; mixed.

vi) Fine S: fraction with particles of 75-150 um in diameter; mixed.

vii) Flowable S: particles 1-2 um in diameter; mixed.

viii) Fine S: fraction with particles of 45-53 um in diameter; mixed.

The soils were wetted to 90% field capacity and the containers were incubated in the dark at a constant 24°C for 3, 6 and 12 weeks. After seven days of incubation, Bentonitic S (C) (treatment iv) was "cultivated", while wet, by rapidly stirring the top 8 to 9 cm of the soil.

Each treatment was repeated nine times for each soil allowing for removal of triplicate samples at each sampling date. After removal, the samples were spread and air dried at room temperature.

3.3.1.3 Experiment Three

This experiment utilized the Breton soil described in Table 1. Samples of 930 g were weighed into 1000 ml plastic containers and treated with one of the following nine treatments:

i) Control: no sulphur added.
ii) Sodium sulphate.
iii) Flowable S.
iv) Bentonitic S (B)
v) Bentonitic S (C): granules of 3-4 mm diameter.
vi) Bentonitic S (C): granules of 1.5-2 mm diameter.
vii) Urea S (C).
viii) Fine S: fraction with particles of 75-150 um diameter.
ix) Fine S: fraction with particles of 45-53 um diameter.

The sulphur treatments were either mixed or banded into the soil at 100 ug g^{-1}. Further, these mixed and banded treatments had a cultivation treatment superimposed to create the treatment-placement-cultivation matrix represented in Table 4.

Samples were wetted to 90% field capacity and the containers were incubated in the dark at a constant 24°C for 3, 6 and 12 weeks. After seven days of incubation, the designated cultivation samples were "cultivated" while moist by rapidly stirring the top 8 to 9 cm of the soil ten vigorous clockwise rotations with a spatula.

Each treatment-placement-cultivation combination was repeated nine times to allow for removal of triplicate samples at each sampling date. After removal, the samples were spread and air dried at room temperature.

Table 4. Treatment-placement-cultivation matrix for Experiment Three.

Treatments	Not Cultivated	Cultivated
i) Control	Mix Band	Mix Band
ii) Sodium Sulphate	Mix Band	Mix Band
iii) Flowable S	Mix Band	Mix Band
iv) Bentonitic S (B)	Mix Band	Mix Band
v) Bentonitic S (C): granules of 3-4 mm diameter	Mix Band	Mix Band
vi) Bentonitic S (C): granules of 1.5-2 mm diameter	Mix Band	Mix Band
vii) Urea S	Mix Band	Mix Band
viii) Fine S: 75-150 um diameter	Mix Band	Mix Band
ix) Fine S: 45-53 um diameter	Mix Band	Mix Band

3.3.2 Effect of Placement and Cultivation on Elemental Sulphur Oxidation

A laboratory incubation experiment was conducted to directly investigate the effects of the interaction between methods of application and cultivation on elemental sulphur oxidation.

Samples of Breton (465 g), Ellerslie (375 g) and Glendon (400 g) soils were weighed into 500 ml plastic containers and treated with 200 ug g^{-1} S of Flowable S (diluted by water in a 50:1 ratio), delivered by pipette, the following ways:

i) Control: no sulphur added.
ii) Placed.
iii) Banded.
iv) Surface,
v) Mixed.

After treatment each sample was wetted to 90% field capacity. The containers were then incubated in the dark at a constant 24°C for 18 and 30 days.

Each treatment was repeated 12 times, for each soil, with one-half (six) being "cultivated" after eight days of incubation. This allowed for removal of three cultivated and three uncultivated treatment samples at each of the sampling dates.

After eight days of incubation all the containers had their lids removed and the soil was allowed to dry for one day. This reduced the soil moisture to approximately 50% field capacity allowing easy removal and thorough "cultivation" of designated samples on a plastic sheet. The samples were then rewetted to 90% field capacity and returned to the incubation chamber.

At sampling dates the removed samples were quickly spread and dried at room temperature.

3.3.3 Effect of Temperature on Elemental Sulphur Oxidation

An incubation study was conducted to investigate the effect of temperature on the rate of oxidation of elemental sulphur.

Samples of Breton (465 g), Ellerslie (375 g) and Glendon (400 g) soils were weighed into 500 ml plastic containers and treated with 200 ug g^{-1} of Fine S (34-45 um diameter fraction) which was thoroughly mixed into the entire soil depth.

After treatment, each sample was wetted to 90% field capacity and then incubated in the dark for 9, 18 and 30 days at 6, 12, 18, 24 and 36°C. For each temperature there were nine elemental sulphur treated containers and nine control containers incubated for each soil. This allowed removal of three treated samples and three corresponding controls at each sampling date.

Removed samples had their lids removed and were placed in the 36°C chamber so that the samples became air dry within 24 hours. Once dry, the samples were ground to pass a 2 mm sieve in preparation for chemical analysis.

3.3.4 Effect of Moisture on Elemental Sulphur Oxidation

A laboratory incubation was conducted to investigate the effect of several moisture regimes on the oxidation of elemental sulphur.

Samples of Breton (465 g), Ellerslie (376 g) and Glendon (400 g) were weighed into 500 ml plastic containers and treated with 200 ug g^{-1} of Fine S (45-53 um diameter fraction) which was thoroughly stirred throughout the entire soil depth.

For each soil, nine treated samples and nine control samples were wetted with deionized water to each of the following moisture levels:

i) 40% field capacity.

ii) 65% field capacity.

iii) 90% field capacity.

iv) 120% field capacity.

Once wetted, each sample was covered and weighed every three days to maintain the initial weight through addition of deionized water. The containers were incubated in the dark at a constant 24°C for 9, 18 and 30 days. At each sampling date three treated samples and three corresponding controls were removed for each of the soil moisture levels. Removed samples had their lids removed and were placed in a 36°C chamber so that samples became air dry within 24 hours. Once dry, the samples were ground to pass a 2 mm sieve in preparation for sulphate analysis.

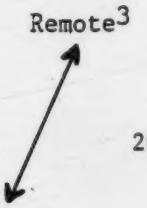
3.3.5 The Effect of Antecedent Agronomic Applications of Elemental Sulphur on the Rate of Oxidation of Subsequent Additions of Elemental Sulphur

An incubation experiment was conducted to investigate the effects of previous field applications of agronomic rates of elemental sulphur on the rate of sulphur oxidation in a subsequent addition under laboratory conditions.

Bulk soil samples were taken from existing field plots at Breton, Ellerslie, Canwood and Canwood Brome sites as illustrated in Fig. 1; at each location a bulk sample was obtained from four sub-plots previously treated with agronomic rates of elemental sulphur (hereafter referred to as S^o soil). A bulk sample was also obtained from the four field plot controls that had never received elemental sulphur (referred to as Nil soil). Additionally, a bulk sample was taken from an area that had never received elemental sulphur and located 200 to 300 meters from the plot (referred to as Remote soil). From the Canwood Brome site a non-cultivated soil with grass cover was sampled, and the samples placed into 500 ml plastic containers in the field (referred to as Virgin soil). These field moist samples were then covered and transferred for storage in a cold room at 4°C for two months. Sampling instruments were cleaned with water and toluene after each sample was taken, in order to minimize the possible transfer of soil microorganisms.

The field moist bulk soil samples, excluding the Canwood Brome Virgin, were passed through a 7 mm screen and placed in aerated 20 litre plastic pails. After sieving of each bulk sample, all sieves and tools were washed with water followed by toluene. The sieved soils were then stored in a cold room at 4°C for two to three months.

Twenty-four 300 g samples (air dry basis), from each field moist bulk sample, were weighed into 500 ml plastic containers. One-half (12) of the samples were treated with 100 ug g^{-1} Fine S and the other 12 were untreated, thus allowing for the removal of



200-300 m

	S^{o1}		Nil[2]
	Nil	S^{o}	
Nil			S^{o}
S^{o}		Nil	

[1] S^{o} - Previously exposed to elemental S.

[2] Nil - Never exposed to elemental S.

[3] Remote - Never exposed to elemental S.

Figure 1. A generalized representation of field plots from which bulk samples were removed for incubation (experiment 3.3.5).

three elemental sulphur treated and three control containers at each of the four sampling dates.

Because the weights of the Canwood Brome Virgin samples varied, the amount of elemental sulphur added was adjusted so that 100 ug g^{-1} S of Fine S was added as a surface application. In all other samples the Fine S was mixed into the soil.

The containers were incubated in the dark at 24°C for 3, 9, 18 and 30 days. After removal, each sample was spread and air dried at room temperature.

3.4. FALLOW FIELD EXPERIMENTS

Sulphur sources were surface applied to the fallow field experiments and mixed through the surface 10 to 12 cm by rotary cultivator (referred to as rotovator). Subsequent cultivation (tillage) operations were done in the same way. Plant growth was controlled by hand weeding and spraying with the herbicides Round Up (Glyphosphate) or Gramoxone (Paraquat).

Soil samples for 0-15 cm, 15-30 cm, 30-60 cm and 60-90 cm were taken with a variety of tools:

i) "Step" sampler; a 3.8 cm diameter coring tube commonly used for the 0-15 cm depth.

ii) "Oakfield" sampler; a 2.0 cm diameter coring tube commonly used for the 15-30 cm depth and to a lesser degree the 0-15 cm and 30-60 cm depths.

iii) Hydraulic truck sampler; a 4.8 cm diameter coring tube used for all depths when samples were required from the 60-90 cm depth.

At each sampling time, 7 to 10 cores were taken and bulked for each subplot. The samples were then air dried and ground through a 2 mm sieve and analyzed for SO_4-S.

3.4.1 Oxidation of Incorporated Elemental Sulphur

Fallow field incubation plots were established in the spring of 1982 at the Breton, Canwood, Ellerslie and Glendon sites (see Table 2) to determine the rate of oxidation of elemental sulphur under field conditions.

Each plot was designed as a randomized complete block with two treatments and four replicates. The treatments were a control and a 50 kg ha^{-1} application of Fine S mixed (tilled) to a depth of 10 cm by rotovator. Each subplot was 2.74 m by 3.05 m and the plots were sampled periodically over the next year.

The plots were tilled a second time after the first sampling.

3.4.2 Effect of Time of Application on the Oxidation of Elemental Sulphur Sources

Fallow field plots were established in May 1980 at the Breton and Ellerslie sites (see Table 2) in order to determine the effect of time of application of elemental sulphur sources on their subsequent availability as sulphate.

Each plot was designed as a split plot with four replicates and four treatments:

i) Control; no sulphur added.

ii) Fine S.

iii) Urea S (A).

iv) Bentonitic S (A).

Subplots were 6.10 m by 6.89 m and early in May 1980 they were split in half so that one-half received an "early" application and the other half received a "late" application approximately three weeks later. All sulphur treatments were applied at 30 kg ha^{-1} and mixed into the soil to a depth of 10 to 12 cm.

Soil samples were taken in mid-summer and in the fall so that rates of oxidation could be determined.

3.4.3 Effect of Tillage on the Availability of Elemental Sulphur Sources

Two fallow field experiments were conducted to investigate the effects of field cultivation on the rate of oxidation of some elemental sulphur sources.

3.4.3.1 Tillage Plot One

Fallow field plots were established in June 1979 at the Canwood and Ellerslie sites (see Table 2). Each plot was initially designed as a randomized complete block with four treatments:

i) Control; no sulphur added.

ii) Sodium sulphate.

iii) Fine S.

iv) Bentonitic S (A).

Ellerslie had four replicates while Canwood had three. All sulphur treatments were applied at 30 kg ha^{-1} and mixed into subplots 3.05 m by 6.89 m. The Ellerslie plot's first soil sampling

occurred on May 10, 1980, while at Canwood the date was June 29, 1980. After the first sampling, one-half of each subplot was tilled to a depth of 10 to 12 cm by rotovator. This tillage operation gave a split plot with individual subplots of 3.05 m by 3.45 m and allowed for a comparison of oxidation of elemental sulphur under tilled and non-tilled conditions. An additional tillage operation was completed approximately two weeks after the first.

A second sampling was completed on July 18 and August 14, 1980, at the Ellerslie and Canwood sites, respectively. Samples were taken from the tilled and untilled portions of each treatment subplot.

3.4.3.2 Tillage Plot Two

Fallow field plots were established in October 1980 at the Breton, Canwood and Glendon sites (see Table 2) to determine the effect of field tillage operations on elemental sulphur oxidation.

Each plot was designed as a split plot with four replicates and four treatments:

i) Control; no sulphur added.

ii) Sodium sulphate.

iii) Fine S.

iv) Urea S (B).

Sulphur was applied at 30 kg ha^{-1} and mixed into subplots 6.10 m by 6.89 m. The subplots were then divided in half with one-half being designated as the tillage portion and the other as the untilled portion. The tillage portions were tilled a second time

during plot establishment and approximately every six to eight weeks, as weather and time allowed, thereafter.

The plots were soil sampled in early May 1981 and in the fall of 1981.

3.5 CROPPED FIELD EXPERIMENTS

3.5.1 Effect of Time of Application of Elemental Sulphur on Its Availability to Barley

A cropped field experiment was established at the Glendon site in 1981 to investigate the effect of time of spring application of elemental sulphur on yield and uptake of sulphur by barley.

The plot was designed as a randomized complete block with four replicates and nine treatments:

i) Control; no nitrogen or sulphur applied.
ii) Urea; early.
iii) Urea; late.
iv) Urea and sodium sulphate; early.
v) Urea and sodium sulphate; late.
vi) Urea and Urea S; early.
vii) Urea and Urea S; late.
viii) Urea S (B); early.
ix) Urea S (B); late.

Early application of nitrogen and sulphur occurred on May 1 while late application occurred just prior to seeding on May 23. Rate of urea nitrogen was 112 kg ha^{-1} and the rate of sulphur was 11.2 kg ha^{-1}, except for treatments (viii) and (ix) where Urea S was used to supply all the 112 kg N ha^{-1} and in doing so supplied 70 kg S ha^{-1} as elemental sulphur.

Fertilizer materials were broadcast and incorporated to 10 cm at the time of application. Blanket applications of K at 120 kg ha^{-1} as KCl and P at 80 kg ha^{-1} as triple superphosphate were applied during the late (May 23) treatment application. A second tillage of the entire plot incorporated this blanket application just prior to seeding the barley.

The plot was harvested in mid-September with two rows 5.03 m in length being removed as the subplot samples. Straw, grain and total yields were determined. Analytical determination of total N and S in the grain and straw was performed, and from these determinations S uptake and N:S ratios were calculated.

3.5.2 Residual Availability of Antecedent Applications of Elemental Sulphur to Barley

The cropped field experiment described in section 3.5.1 was maintained through the 1982 growing season to investigate the residual availability of elemental sulphur to barley.

Urea (112 kg N ha^{-1}) was surface applied to each subplot, excluding the control, and incorporated to 10 cm on May 29. Barley was then sown and harvested in late September by removing two rows 5.03 m in length as subplot samples from all treatments, excluding (ii) as it was redundant. Straw, grain and total yields were determined and analyses for total N and S were performed and from these determinations S uptake and N:S ratios were calculated.

3.5.3 Availability of Surface Applied Elemental Sulphur to Bromegrass

Field plots were established in June 1981 on a mature bromegrass (Bromus inermis) stand at the Canwood Brome site (see Table 2) to determine the availability of surface applied elemental sulphur to bromegrass.

The plot was designed as a randomized complete block with four replicates and six treatments:

i) Control; no nitrogen or sulphur applied.

ii) Urea.

iii) Sodium sulphate.

iv) Urea and sodium sulphate.

v) Urea and Urea S (B) 1981; Urea and Urea S (C) 1982 and 1983.

vi) Urea and Fine S.

Fertilizers were surface applied with N at 112 kg ha^{-1} (as urea) and S at 11.2 kg ha^{-1} to subplots 3.05 m by 6.10 m. Spring fertilization of the plot was repeated in 1982 and 1983.

In the fall of each year (also mid-summer of 1981 and 1982), three harvest quadrants (totalling 1.12 m^2) were removed from each subplot and treatment yields were determined. Analyses for total N and S were performed and from these determinations S uptake and N:S ratios were calculated.

3.6 ANALYTICAL PROCEDURES

The soluble sulphate content of soil samples was measured by extraction with 0.1 M $CaCl_2$ followed by determination of the sulphate content of the extract by the method of Johnson and Nishita

(1952) as modified by Dean (1966). The total sulphur content of plant material was determined by the method of Tabatabai and Bremner (1970). The total nitrogen content of plant material was determined by the semi-micro-Kjeldahl method of Bremner (1965). For soil characterization, total N (excluding nitrates) was determined by colorimetric analysis with a Technicon Auto-Analyser II system (Technicon 1979).

Soil pH was measured with a pH meter using a glass electrode in a suspension of 1:2.5 soil to water ratio (McKeague 1978).

An estimate of field capacity moisture content (approximately -33 kPa potential) for the soils used in the laboratory incubation experiments was made by placing 250 cc of soil in 3.8 cm diameter plastic cylinders and adding sufficient water to wet approximately three-quarters of the soil depth. The tubes were covered (but not sealed) to minimize evaporation yet allow free drainage. After 48 hours the 3.8 to 6.4 cm depth of soil was sampled for moisture determination.

Bulk density was estimated by taking two replicates consisting of three cores (4.8 cm diameter) bulked for the 0-15 cm, 15-30 cm, 30-60 cm and 60-90 cm depths. The soil samples were dried at 105°C for 24 hours and weighed. Bulk density was calculated from the ratio of soil weight (Mg) to volume of the core (m^3).

At each laboratory incubation sampling date, the elemental sulphur treated soils and untreated control soils were analyzed for sulphate-S to evaluate the soils' ability to oxidize elemental sulphur. The apparent net recovery (%) as sulphate-S was then

calculated, in the following manner, and tabulated:

$$\frac{\text{ug g}^{-1}\ SO_4\text{-S in elemental sulphur treated soil} - \text{ug g}^{-1}\ SO_4\text{-S in untreated control soil}}{\text{ug g}^{-1}\ \text{elemental sulphur added}} \times 100$$

$$= \text{apparent net recovery \%}$$

For the field experiments the amount (kg ha^{-1}) of sulphate found in the soil sample depth was determined and the same calculation used for determination of the apparent net recovery (%).

All soil sulphate sulphur, yield and plant composition data were subjected to an analysis of variance. Differences between means were confirmed using the method of least significant difference (LSD) at the 5% level of probability (Steel and Torrie 1977, Hill and Little 1978).

4. RESULTS AND DISCUSSION

The several experiments comprising this study will be discussed under the same headings used in the Materials and Methods section. Each separate Laboratory Incubation Experiment will be discussed individually while general discussions will follow the Fallow Field Experiments and the Cropped Field Experiments. A summary discussion of the total results will conclude this section.

4.1 LABORATORY INCUBATION EXPERIMENTS

4.1.1 Oxidation of Elemental Sulphur as Affected by Source, Method of Application and Cultivation

In three experiments the recovery of sulphate from elemental sulphur was markedly affected by sulphur source and method of application. In an initial experiment, "cultivation" gave increased recovery of sulphate from one elemental sulphur source but not from the other sulphur sources. In a later experiment, a less vigorous "cultivation" appeared to have no effect on oxidation.

4.1.1.1 Experiment One

The Ellerslie soil oxidized significantly more elemental sulphur than did the Canwood soil (Table 5). Overall average recovery of sulphate from elemental sulphur after 12 weeks (48%) of incubation was significantly greater than at 3 weeks (31%). Recovery of sodium sulphate was complete at both sampling dates while mixed Fine S and "cultivated" Urea S (A) recovery was approximately 40%

Table 5. Apparent net recovery (%), as sulphate-S, of applied (25 ug g^{-1}) fertilizer-S as affected by sulphur source, method of application and "cultivation". Incubation Experiment One.

Treatment	Method of Application	Net % Recovery of S as SO_4-S						
		Canwood Soil		Ellerslie Soil		Sampling Date - Treatment Means[a]		Cumulative Treatment Means[b]
		3 wks	12 wks	3 wks	12 wks	3 wks	12 wks	
Fine S	Mixed	38.2	76.2	36.6	81.4	37.4	78.8	58.1
Fine S	Placed	14.6	14.4	27.0	46.4	20.8	30.4	25.6
Na_2SO_4	Mixed	96.4	109.2	99.6	106.8	98.0	108.0	103.0
Bentonitic S (A)	Placed	4.8	20.0	1.4	17.4	3.1	18.7	10.9
Bentonitic S (A)	Mixed	3.6	10.8	23.4	19.6	13.5	15.2	14.4
Urea S (A)	Mixed	5.8	16.2	26.4	12.2	16.1	14.2	15.2
Urea S (A)	Mixed and "cultivated"	38.8	78.4	45.2	115.0	42.0	96.7	69.4
Urea S (A)	Placed	6.6	14.8	20.6	25.8	13.6	20.3	17.0
Soil Means[c]		34.3		44.1				
Sampling Means[d]						30.6	47.8	

[a] LSD at 5% level is 20.4 to compare sampling date - treatment means.

[b] LSD at 5% level is 14.4.

[c] Soil means are significantly different as determined by F test.

[d] Sampling date means are significantly different as determined by F test.

at 3 weeks and 80 to 100% at 12 weeks. Recovery of incorporated Bentonitic S (A) and Urea S (A) averaged roughly 15%. When compared to mixing, placement of Fine S significantly reduced recovery while placement of Urea S (A) and Bentonitic S (A) did not signifi- cantly affect oxidation. "Cultivation" of Urea S significantly increased sulphate recovery.

4.1.1.2 Experiment Two

The average amount of elemental sulphur oxidized by the Breton (41%) and Ellerslie (46%) soils was not significantly different (Table 6). Average elemental sulphur recovery after 6 weeks (52%) was significantly greater than at 3 weeks (32%) but not significantly different from the recovery at 12 weeks (46%).

Recovery of sodium sulphate ranged from approximately 75 to 100%, but averaged approximately 90%. The recovery of mixed and "cultivated" Bentonitic S (C) was low (<5.0%) for all three sampling dates while the recovery of crushed (<150 um) Bentonitic S (C) was 55% and was equal to or greater than the recoveries from Flowable S (50%) and the two Fine S fractions (47 and 60%).

4.1.1.3 Experiment Three

"Cultivation" of the Breton soil one week after application of elemental sulphur did not significantly increase sulphur oxidation (Table 7). Average recovery at 6 weeks (33%) was significantly greater than at 12 weeks (26%) and both were greater than recovery at 3 weeks (19%). Bentonitic S (B) and Bentonitic S (C) showed little

Table 6. Apparent net recovery (%), as sulphate-S, of applied (100 ug g^{-1}) fertilizer-S as affected by sulphur source and "cultivation". Incubation Experiment Two.

Treatment	Method of Application	Net % Recovery of Applied S as SO_4-S									
		Breton Soil			Elleralie Soil			Sampling Date - Treatment Means[a]			Cumulative Treatment Means[b]
		3 wks	6 wks	12 wks	3 wks	6 wks	12 wks	3 ks	6 wks	12 wks	
Sodium sulphate	Mixed	86.7	98.8	93.8	73.0	105.7	75.8	79.9	102.3	84.8	89.0
Bentonitic S (C)	Mixed	0.9	0.3	2.1	2.5	0.9	2.6	1.7	0.6	2.4	1.6
Bentonitic S (C)	Mixed and "cultivated"	0.6	3.8	2.2	0.0	5.1	2.2	0.3	4.5	0.0	1.6
Bentonitic S (C) crushed to <150 um	Mixed	42.7	52.8	60.8	51.4	78.7	43.4	47.1	65.8	52.1	55.0
Fine S (75-150 um)	Mixed	27.5	47.4	57.8	21.8	56.5	68.4	24.7	52.0	63.1	46.6
Flowable S (1-2 um)	Mixed	26.1	54.8	59.3	39.5	62.5	59.9	32.8	58.7	59.6	50.4
Fine S (45-53 um)	Mixed	26.7	53.8	56.8	46.9	110.6	68.4	36.8	82.2	62.6	60.5
Soil means[c]			40.7			46.3					
Sampling means[d]								31.9	52.3	46.4	

[a] LSD at 5% level is 17.6 to compare sampling dates - treatment means.

[b] LSD at 5% level is 18.2.

[c] Soil means are not significantly different as determined by F test.

[d] LSD at 5% level is 10.8.

Table 7. Apparent net recovery (%), as sulphate-S, of applied (100_4ug g^{-1}) fertilizer-S as affected by sulphur source, method of application and "cultivation". Incubation Experiment Three.

Treatment	Method Applied	Net % Recovery of Applied S as SO_4-S							
		Breton Soil							
		3 weeks		6 weeks		12 weeks		Mixed and Banded Means[a]	Cumulative Means[b]
		Not cult.	Cult.	Not cult.	Cult.	Not cult.	Cult.		
Na_2SO_4	Mixed	87.0	88.8	98.8	98.7	93.8	78.6	91.0	
	Banded	85.7	82.8	89.8	113.2	96.3	75.6	90.6	90.8
Flowable S (1-2 um)	Mixed	26.1	38.8	53.8	60.7	43.6	56.6	46.6	
	Banded	12.7	15.8	14.6	21.7	21.8	40.0	21.1	33.9
Bentonitic S (B)	Mixed	1.3	0.2	0.0	4.3	2.8	1.2	1.6	
	Banded	0.6	1.6	-0.4	-0.1	1.7	1.4	0.8	1.2
Bentonitic S (C) (1.5-2 mm)	Mixed	0.8	1.2	1.7	-0.8	0.9	1.5	0.9	
	Banded	0.1	1.5	0.4	3.1	1.4	0.7	1.2	1.1
Bentonitic S (C) (2-4 mm)	Mixed	0.9	-0.3	0.3	3.7	2.1	-0.5	0.9	
	Banded	0.9	-2.1	-1.4	-1.6	2.7	-0.9	-0.4	0.3
Urea S (C)	Mixed	1.4	5.2	1.1	12.4	3.3	9.6	5.5	
	Banded	1.4	8.8	2.4	9.2	2.0	22.0	7.6	6.6
Fine S (75-150 um)	Mixed	27.5	31.8	47.3	60.1	57.8	54.6	46.5	
	Banded	7.0	5.6	5.7	3.7	12.4	8.0	7.1	26.8
Fine S (45-53 um)	Mixed	26.0	41.2	54.7	59.9	56.8	53.0	49.1	
	Banded	4.3	8.6	0.9	9.5	9.4	15.1	8.0	28.6
Sampling Date Means[c]		18.6		33.3		25.8			

[a] LSD at 5% level is 8.9.
[b] LSD at 5% level is 6.9.
[c] LSD at 5% level is 3.8.

or no oxidation whether mixed or banded. Urea S (C) also gave low recovery (1-22%) with no significant effect of banding.

Banding of the finely divided elemental sulphur materials significantly reduced oxidation. Average recovery of the banded fractions of Fine S was reduced by a factor of approximately 6.5 when compared to the mixed fractions, while average recovery of banded Flowable S was reduced by a factor of 2.2. Thus, the recovery from banded Flowable S (21%) was significantly greater than recovery from the banded Fine S fractions (7-8%).

4.1.1.4 Discussion

Two procedural aspects of these experiments require illumination so that a coherent discussion may follow. "Cultivation" in Experiment One involved removal of the soil sample followed by thorough mixing on a plastic sheet while "cultivation" in Experiment Three involved a vigorous stirring of the wet soil. Thus, cultivation in Experiment One was much more complete than in Experiment Three. Secondly, the Urea S (A) used in Experiment One contained 10.4% sulphur while Urea S (C) in Experiment Three contained 20.0% sulphur.

Elemental sulphur oxidation rates are proportional to the exposed surface area of the applied sulphur (Fox et al. 1964). Thus, as the diameter of the applied particles or pellets increases, the exposable surface area decreases thereby reducing potential oxidation rates.

Placement of Fine S (Experiment One, Table 5) reduced the average oxidation from 58 to 26% (a factor of approximately 2.2).

This reduction can be attributed to a decrease in Fine S surface area exposed to soil contact. Similarily, the low recovery of sulphate from the mixed and placed Urea S (A) can be explained by a decrease in the exposed surface area of the elemental particles (mean diameter of 25 um) that impregnate the urea pellet. When Urea S is added to soil, the urea dissolves leaving an aggregation or localized "placement" of fine elemental sulphur particles approximately the same size as the original pellet. This results in a 40-fold reduction in the exposed surface area (Janzen 1984) as only the outermost particles are exposed to microbial oxidation. Additionally, as the elemental sulphur content of the urea pellet increases, the more pronounced this effect will become. Since Urea S (C) contains 20% sulphur and Urea S (A) contains 10.4% sulphur, theoretically mixed Urea S (C) would oxidize more slowly than mixed Urea S (A).

Observation of the poor recoveries from the Bentonitic S products (Nyborg 1980, Noellemeyer et al. 1981, Ukrainetz 1982) can be explained in a similar way. With time, Bentonitic S becomes fractured into fine particles thereby greatly increasing the potentially exposable surface area of the elemental sulphur. If these bentonitic sulphur deposits are not disrupted and mixed throughout the soil, the exposed surface area remains low and the oxidation potential is small. This could also explain the comparatively low recoveries of Bentonitic S in this study. The high recoveries from "cultivated" Urea S (A) in Experiment One was apparently a result of an effective disruption of the elemental

sulphur "placements" and subsequent thorough mixing of the sulphur particles throughout the soil. These results are similar to those observed by Janzen (1984) with Urea S in a potted canola experiment.

A less thorough cultivation in Experiment Three did not increase the oxidation of Urea S (C) or the Bentonitic S materials presumably because the elemental sulphur aggregations were not disrupted and mixed through the soil. This is consistent with field and laboratory observations (Nyborg and Solberg 1985; unpublished) of cultivated and banded Urea S (C) in the field and cultivated Urea S (C) and Bentonitic S (B and C) in the laboratory. In the field, Urea S (C) "pellets" could still be found four months after they had been incorporated and subsequently cultivated by a high speed rotorary cultivator. Similarily, bands of Urea S (C) could easily be found even after rotorary cultivation across the band. It would appear that a single field cultivation may not be intense enough for disruption and thorough mixing of the Urea S elemental sulphur particles through the soil. In the laboratory, aggregations of fine elemental sulphur could often be found in the "cultivated" Bentonitic S treatments indicating that "cultivation" had not been intense enough to disperse these aggregations or that the pre- "cultivation" incubation period was not of sufficient length to allow for fracturing of the granules. High recovery of sulphate from the crushed Bentonitic S (C) in Experiment Two indicates that this material may give high recoveries if the aggregations of fractured elemental sulphur particles can be adequately dispersed.

In Experiment Three, banding elemental sulphur reduced oxidation since band application reduces the surface area exposure to

microbial contact. Banded Flowable S recovery was significantly greater than recovery from the banded Fine S fractions. Flowable S (1-2 um particle diameter), in a 50:1 water dilution, was added to the soil by pipette. This method of application gave bands that were approximately 8 mm wide while the Fine S bands were 4 to 5 mm wide. These relatively disperse Flowable S bands, containing smaller elemental sulphur particles, allowed for greater surface area exposure and consequently greater sulphate recovery.

In Experiment Three, the average recovery of elemental sulphur after 12 weeks was significantly less than at 8 weeks. This can be attributed to microbial immobilization of the sulphate liberated during oxidation of elemental sulphur.

4.1.2 Effect of Placement and Cultivation on Elemental Sulphur Oxidation

This experiment was undertaken to further examine the effect of various methods of elemental sulphur application and cultivation on subsequent oxidation.

The average recovery of elemental sulphur from the Ellerslie (61%) and Glendon (64%) soils were significantly greater than from the Breton (28%) soil (Table 8). Only the Glendon soil had a significantly higher recovery of sulphate after 30 days (72%) than at 18 days (56%). The average recovery for both sampling dates was approximately 28 and 61% for the Breton and Ellerslie soils, respectively.

Cultivation gave significantly greater recoveries of elemental sulphur for all three soils. Cultivation of banded and

Table 8. Apparent net recovery (%), as sulphate-S, of applied (200 ug g^{-1}) elemental sulphur, from incubated soils, as affected by placement and "cultivation".

		% Recovery as SO_4-S							
		18 Days		30 Days		Means		Cumulative Treatment Means[a]	Soil Means[b]
Soil	Treatments	Not Cult.	Cult.	Not Cult.	Cult.	Not Cult.	Cult.		
Breton	Placed	8.5	21.6	5.7	32.3	7.1	27.0	17.1	
	Banded	10.8	35.0	6.6	31.9	8.7	33.5	21.1	
	Surface	29.8	39.8	28.1	31.8	29.0	35.8	32.4	
	Mixed	38.2	37.5	38.6	47.9	38.4	42.7	40.6	
Treatment-Cult. Means[c]		21.8	33.5	19.6	36.0	20.8	34.8		
Sampling Date Means		27.7		27.9					27.8
Ellerslie	Placed	31.0	47.5	26.4	62.9	28.7	55.2	42.0	
	Banded	35.3	50.3	28.2	57.6	31.8	54.0	42.9	
	Surface	59.1	86.0	65.8	88.3	62.5	87.2	74.8	
	Mixed	75.8	95.4	80.4	80.3	78.1	87.9	83.0	
Treatment-Cult. Means		50.3	69.8	50.2	72.3	50.3	71.1		
Sampling Date Means		60.1		61.3					60.7
Glendon	Placed	22.0	62.2	35.4	77.2	28.7	69.7	49.2	
	Banded	35.1	58.3	50.1	72.9	42.6	65.6	54.1	
	Surface	43.7	68.6	78.5	89.4	61.1	79.0	70.1	
	Mixed	74.9	82.7	74.8	96.2	74.9	89.5	82.2	
Treatment-Cult. Means		43.9	68.0	59.7	83.9	51.8	76.0		
Sampling Date Means[d]		56.0		71.8					63.9

[a] LSD at 5% is 10.1, 20.3, 12.7 for Breton, Ellerslie and Glendon, respectively.
[b] LSD at 5% level is 6.4.
[c] LDS at 5% is 7.1, 14.4, 9.0 for Breton, Ellerslie and Glendon, respectively.
[d] Glendon sampling date means are significantly different as determined by F test.

placed treatments frequently resulted in considerably larger increases in sulphur recovery than did cultivation of the surface and mixed treatments.

For all three soils, the surface and mixed treatments gave significantly greater recoveries of sulphur than did the placed and banded treatments. There was not a significant difference between surface and mixed treatments or between placed and banded treatments.

As discussed earlier in Section 4.1.1.4, any method of application (or material applied) that restricts the elemental sulphur surface area exposure will also reduce the rate of oxidation. Thus, even though Flowable S is made up of particles 1-2 um in diameter, placement and banding restricts the exposure of these particles to microbial oxidation. The thorough method of "cultivation" utilized in this experiment effectively disrupts the placements and bands thereby mixing the elemental sulphur particles throughout the soil and increasing oxidation.

Surface application gave surprisingly high levels of recovery of sulphate. Surface application was thought to reduce exposure of the sulphur particles to soil contact and thus reduce recovery. A possible explanation is that the Flowable S, which was evenly spread over the surface of the dry soil, was carried into soil voids by watering, thereby increasing the elemental sulphur exposure to microbial oxidation. In addition, the oxygen and moisture supply at the soil surface inside the covered incubating container may be optimal for sulphur oxidation. Interestingly, in a field study, Bertrand (1973) observed very high rates of oxidation of heavy applications of wind blown sulphur at or near the soil surface.

4.1.3 Effect of Temperature on Elemental Sulphur Oxidation

This experiment was undertaken to evaluate the effect of temperature on sulphur oxidation in three Alberta soils (Table 9 and Fig. 2). The average amount of elemental sulphur oxidized by the Ellerslie soil (61.5%) is significantly greater than the Glendon soil (43.5%) and the Breton soil (32.9%). There are significant differences between all of the sampling dates means for the Ellerslie (LSD = 13.9) and Glendon (LSD = 7.7) soils. The average elemental sulphur recovered from the Breton soil after 30 days (44.5%) was significantly greater than at 18 days (30.9%) and 9 days (23.5%).

The results of this experiment are similar with oxidation rates at 36°C being 6.0 (Breton), 4.8 (Ellerslie) and 4.8 (Glendon) times faster than at 6°C when incubated for 30 days. It would appear that the Breton soil is very sensitive to temperature. At the first sampling date (9 days), the 6, 12 and 18°C temperature treatments gave similar recoveries of elemental sulphur. This trend continued to the second sampling date where the 6 and 12°C treatments gave similar sulphur recovery. By 30 days all of the treatments were significantly different but the 6°C treatment gave the same recovery as it did at 9 and 18 days.

Nor and Tabatabai (1977) reported oxidation rates 2.3 to 6.1 times faster at 30°C than at 4°C when incubated for 74 days. Li and Caldwell (1966) reported oxidation rates being four times faster at 30°C than at 23°C when incubated for 60 days.

Perhaps, after rewetting the dry soil, the portion of the soil microflora responsible for elemental sulphur oxidation requires

Table 9. Apparent net recovery (%), as sulphate-S, of applied (200 ug g^{-1}) elemental sulphur, from incubated soils, as affected by temperature.

Temperature °C	Net % Recovery of Applied S as SO_4-S											
	Breton (Days)				Ellerslie (Days)				Glendon (Days)			
	9	18	30	Temp. Means[a]	9	18	30	Temp. Means	9	18	30	Temp. Means
6[b]	12.2	10.2	12.9	11.8	11.3	14.4	23.0	16.2	12.7	15.9	20.2	16.3
12	10.4	15.3	21.3	15.7	22.3	47.0	55.2	41.5	16.7	22.5	29.0	22.7
18	14.4	27.5	38.7	26.9	35.7	69.8	96.2	67.2	21.0	29.1	48.4	31.7
24	30.9	45.5	71.8	49.4	65.6	81.9	105.9	84.5	41.6	58.1	75.0	58.2
36	49.7	55.8	77.6	61.0	82.1	103.2	109.7	98.3	77.6	91.1	97.6	88.8
Sampling Means[c]	23.5	30.9	44.5		43.4	63.3	78.0		33.9	43.3	53.4	
Soil Means[d]				32.9				61.5				43.5

[a] LSD at 5% is 15.2, 17.9 and 10.0 for Breton, Ellerslie and Glendon, respectively.
[b] To compare sampling dates within a particular temperature or to compare temperature treatments within a particular sampling date, LSD at 5% is 5.6, 5.1 and 6.0 for Breton, Ellerslie and Glendon, respectively.
[c] LSD at 5% is 11.8, 13.9 and 7.7 for Breton, Ellerslie and Glendon, respectively.
[d] LSD at 5% is 11.3.

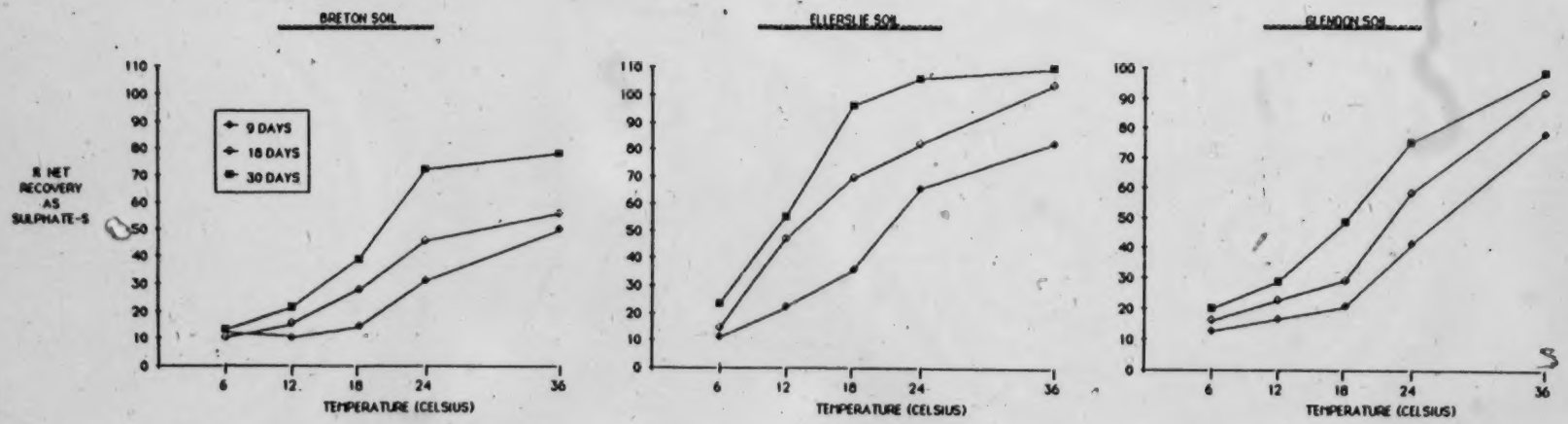


Figure 2. Percent recovery of applied (200 ug g^{-1}) elemental sulphur (45-53 um fraction) from soils incubated at 90% field capacity for 9, 18 and 30 days at 6, 12, 18, 24 and 36°C.

progressively long periods of time, as temperature decreases, for the return of full oxidative capacity. Nor and Tabatabai (1977), in an incubation experiment, observed a lag period in which rewetted air dry soils oxidized less elemental sulphur than wetted field moist samples. Thus, for the Breton soil this lag period may be extended by decreased temperature.

4.1.4 Effect of Moisture on Elemental Sulphur Oxidation

This experiment was undertaken to examine the effect of moisture on sulphur oxidation in three Alberta soils. The average amounts of elemental sulphur oxidized by the Breton (35.2%), Ellerslie (52.5%) and Glendon (46.7%) soils are not significantly different (Table 10, Fig. 3). For all three soils, the sampling date means are significantly different. For the Breton and Glendon soils, the average recovery from the 90% field capacity (F.C.) and 120% F.C. treatments were significantly greater than the average recovery from the 40% F.C. and 65% F.C. treatments. There was not a significant difference between each soil's 90% F.C. and 120% F.C. treatments nor between the 40% F.C. and 65% F.C. treatments. The average Ellerslie moisture treatment recoveries were all significantly different.

Significant quantities of elemental sulphur were oxidized at all moisture levels with the lowest recovery from the 40% F.C. treatment. The 40% F.C. treatment approximates the soil water content at wilting point and at these low levels of soil moisture oxidation is limited by a lack of water for microbial growth.

Table 10. Apparent net recovery (%), as sulphate-S, of applied (200 ug g^{-1}) elemental sulphur, from incubated soils, as affected by soil moisture.

	Net % Recovery of Applied S as SO_4-S											
	Breton (Days)				Ellerslie (Days)				Glendon (Days)			
% Field Capacity	9	18	30	Treat. Means[a]	9	18	30	Treat. Means	9	18	30	Treat. Means
40	8.3	10.6	34.4	17.8	11.2	16.2	32.0	19.8	12.1	23.3	53.2	29.5
65	10.9	17.2	35.1	21.1	28.4	32.9	63.1	41.5	19.6	51.0	70.0	46.9
90	30.9	45.5	71.8	49.4	65.6	81.9	105.9	84.5	41.7	58.1	75.0	58.3
120	37.0	46.2	74.6	52.6	44.5	62.9	84.5	64.0	41.8	58.4	56.2	52.1
Sampling Means[c]	21.8	29.9	54.0		37.4	48.5	71.4		28.8	47.7	63.6	
Soil Means[d]				35.2				52.5				46.7

[a] LSD at 5% is 8.8, 11.1 and 18.0 for Breton, Ellerslie and Glendon, respectively.
[b] To compare sampling dates within a particular moisture level or to compare moisture levels within a particular sampling date, LSD at 5% is 6.9, 6.3 and 8.7 for Breton, Ellerslie and Glendon, respectively.
[c] LSD at 5% is 7.6, 8.6 and 14.0 for Breton, Ellerslie and Glendon, respectively.
[d] Soil means are not significantly different at the 5% level as determined by F test.

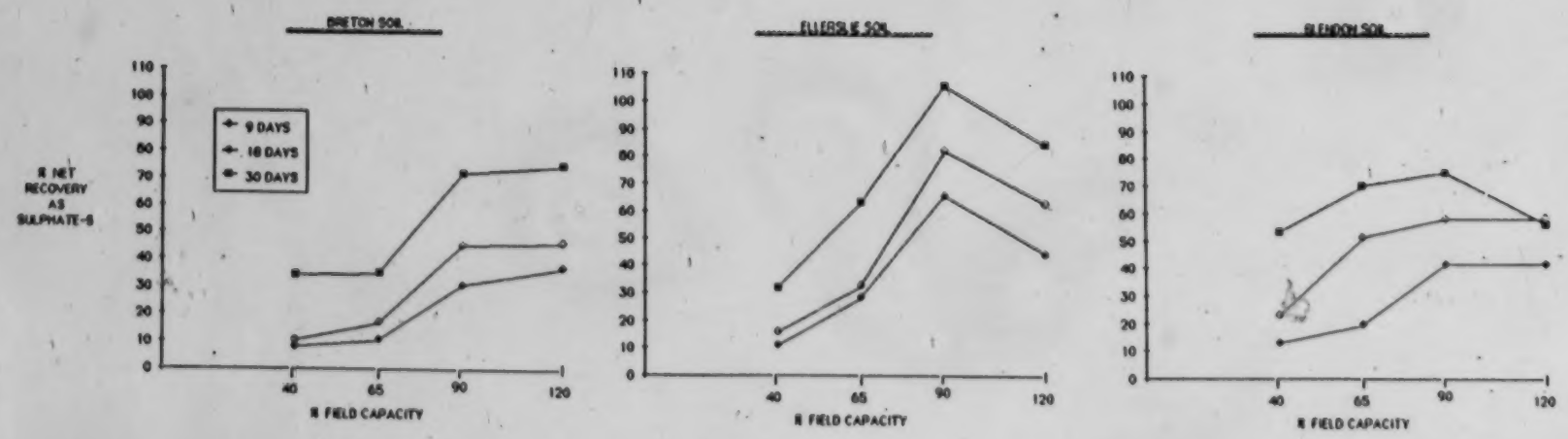


Figure 3. Percent recovery of applied (200 ug g^{-1}) Fine elemental sulphur (45-53 um fraction) from soils incubated at 24°C for 9, 18 and 30 days at 40, 65, 90 and 120% field capacity.

The Breton 65% F.C. treatment also appeared to contain insufficient water to increase oxidation over the 40% F.C. treatment. The 90% F.C. and 120% F.C. Breton treatments resulted in similar recoveries thereby indicating sufficient water and oxygen, at both moisture levels, for maximum oxidation.

The Ellerslie soil showed a parabolic relationship between sulphur recovery and moisture content at all three sampling dates while the Glendon soil tended to be parabolic after 30 days. Maximum oxidation occurred at 90% F.C. with the 65% F.C. treatment intermediate between 90% F.C. and 40% F.C. The 120% F.C. treatment presumably reduced oxidation by reducing the amount of oxygen available to the oxidizing populations.

These results are generally in agreement with the results of Moser and Olson (1953) who found, in a three-week incubation at 25°C, that maximum oxidation occurred near field capacity and that an overall parabolic relationship existed between moisture content and oxidation rate. Janzen (1984) observed a similar parabolic relationship that became more pronounced as temperature was increased stepwise from 3°C to 30°C.

4.1.5 Effect of Antecedent Agronomic Applications of Elemental Sulphur on the Rate of Oxidation of Subsequent Additions of Elemental Sulphur

There were no significant differences between the soil recovery means (Table 11, Fig. 4). Previous soil treatments were significantly different for the Ellerslie, Canwood and Brome soils while treatments were not significantly different for the Breton soil.

Table 11. Apparent net recovery (%), as sulphate-S, of applied (100 ug g^{-1}) elemental sulphur, from incubated soils, as affected by soil moisture.

Antecedent Soil Treatment	Net % Recovery of Applied S as SO_4-S																			
	Breton (Days)					Ellerslie (Days)					Canwood (Days)					Brome (Days)				
	3	9	18	30	Treat. Means[a]	3	9	18	30	Treat. Means	3	9	18	30	Treat. Means	3	9	18	30	Treat. Means
Remote[b]	6.8	11.4	5.4	18.5	10.5	0.3	2.9	10.8	23.9	9.5	0.0	2.3	7.6	29.0	9.7	1.0	2.7	11.2	22.4	9.3
Nil	4.5	6.1	27.3	48.1	21.5	7.5	10.3	27.2	37.1	20.5	2.0	3.0	32.4	54.7	23.0	3.0	11.5	30.3	40.9	21.4
S^o	4.9	16.0	37.2	56.5	28.7	6.7	18.2	36.5	44.9	26.6	5.8	31.0	53.3	73.9	41.0	4.9	29.0	50.9	70.2	38.8
Virgin	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1.3	3.3	7.1	11.1	5.7
Sampling means[c]	5.4	8.8	17.5	30.8		4.8	10.5	24.8	35.3		2.6	12.1	31.1	52.5		2.6	11.6	24.9	36.2	
Soil means[d]					20.2					18.9					24.6					18.8

[a] LSD at 5% is 7.4, 17.9 and 16.9 for Ellerslie, Canwood and Brome, respectively. Breton treatments are not significant as determined by F test.
[b] To compare sampling dates within a particular treatment or to compare treatments within a particular sampling date, LSD at 5% is 1.8, 2.0, 1.8 and 1.7 for Breton, Ellerslie, Canwood and Brome, respectively.
[c] LSD at 5% is 21.7, 8.6, 20.7 and 16.9 for Breton, Ellerslie, Canwood and Brome, respectively.
[d] Soil means are not significantly different at the 5% level as determined by F test.

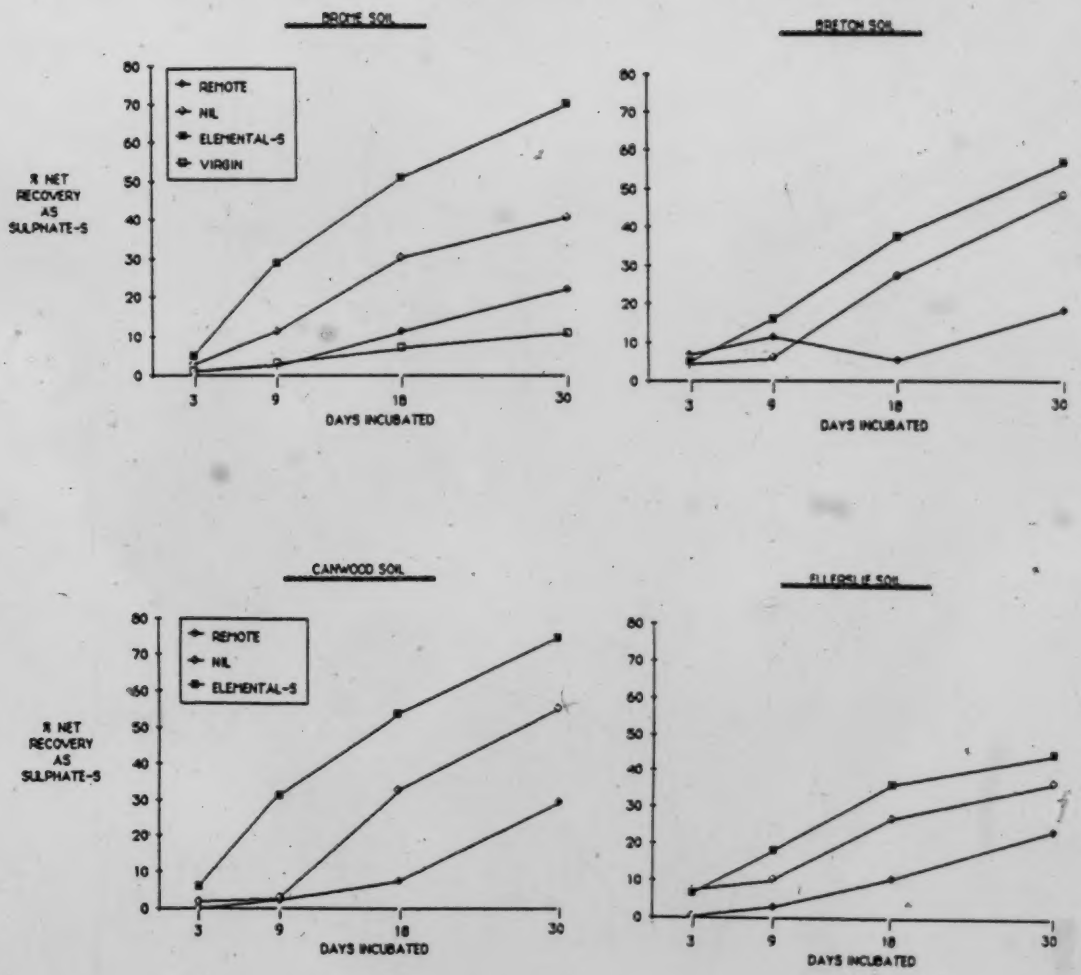


Figure 4. Antecedent elemental sulphur application effect on subsequent % recovery of applied (100 ug g^{-1}) Fine elemental sulphur from soils incubated at 24°C and 90% field capacity for 3, 9, 18 and 30 days.

Sampling date means were significantly different for all four soils. The treatment-time interaction was highly significant for all soils.

The average recovery of the Ellerslie S^o treatment (26.6%) was not significantly different from the average recovery of the Nil treatment (20.5%) but both were significantly greater than the average Remote treatment (9.5%) recovery. The average recovery of the Canwood S^o treatment (41.0%) was significantly greater than the Nil (23.0%) and Remote (9.7%) recovery. Similarily, the average recovery of the Brome S^o treatment (38.8%) was significantly greater than the recoveries from the Nil (21.4%), Remote (9.3%) and Virgin (5.7%) treatments.

The addition of large amounts of elemental S has been shown to greatly increase the numbers of sulphur oxidizers and oxidation rate (Moser and Olson 1953, Bertrand 1973, Wainwright 1978, 1979). The addition of low levels of elemental sulphur was shown to have no measureable effect on subsequent oxidation by Vitolins and Swaby (1969).

This experiment indicates that subsequent additions of elemental sulphur are oxidized more quickly, in the laboratory, by soils previously receiving agronomic levels of elemental sulphur. Soils taken from subplots adjacent to the elemental sulphur enriched soils appear to have an increased oxidative capacity over Remote soils obtained some distance from the field plot (Fig. 1).

The highly significant time-treatment interaction would suggest that the oxidation by the untreated soils lags "behind" the sulphur amended soils. An explanation for this could be that the

elemental sulphur amended soil contains a larger or more effective complement of sulphur oxidizing bacteria. This could also help explain why the adjacent soils appear to also have increased oxidative capacities. During field operations of tillage, sampling and weed control there may be a low level of "inoculation" of these adjacent soils. The Remote soils would not be subjected to this low level "inoculation" as they are sufficiently removed from the plot.

Subsequent additions of elemental sulphur to the enriched soil would be more rapidly oxidized because the soil contains a more vigorous microbial population that is actively oxidizing the previous sulphur application. The adjacent "inoculated" Nil soils would lag behind the enriched soil because the Nil microbial population is not actively oxidizing elemental sulphur at the time of application and a short adjustment period is required. The Remote soil would lag further as it is neither microbially enriched (inoculated) nor actively oxidizing elemental sulphur.

Other more mundane observations from this experiment should be stated. By the final sampling date (30 days), all of the soils had oxidized significant quantities of elemental sulphur. The "Virgin" soil from the Canwood Brome site was beginning to oxidize significant quantities of elemental sulphur even though it had been applied to the grass covered soil surface.

4.2 FALLOW FIELD EXPERIMENTS

4.2.1 Oxidation of Incorporated Elemental Sulphur

This experiment was undertaken to evaluate oxidation of incorporated Fine S and to augment results from previously conducted experiments (sections 3.4.2 and 3.4.3).

Fine S (50 kg ha^{-1}) was broadcast and incorporated into fallow plots, that had never received elemental sulphur before, at four sites in May or June of 1982. Plots were sampled periodically over the next year. Substantial amounts (11 to 42%) of the elemental sulphur could be recovered as sulphate after one or two months (Table 12). Results are presented only for the 0-30 cm layer as virtually no sulphate was found below the 30 cm depth. This indicates little loss of the generated sulphate due to leaching. At Canwood and Glendon each sampling date found more elemental sulphur converted to sulphate. After 115 days at Canwood and 360 days at Glendon, approximately 65% of sulphur was recovered as sulphate. The Breton and Ellerslie plots never had more than 50% of the added elemental sulphur recoverable as sulphate and after one year the levels of sulphate were equal to or slightly less than at the initial sampling (Breton, 32%; Ellerslie, 37%). Because there was little loss to leaching, this would indicate that significant amounts of sulphate were either immobilized or that much of the added sulphur remained as unoxidized elemental sulphur. Freney and Spencer (1960) found rather dramatic immobilization "losses" in incubated soils with large amounts of sulphate added. Koehler and Roberts (1983) observed

Table 12. Apparent net recovery (%), as sulphate-S, of incorporated Fine S applied at 50 kg ha^{-1} in May or June, 1981, to cultivated fallow soils at four sites.

Site	Date Applied	Days of Field Incubation	Net Recovery % in 0-30 cm Depth
Breton	May 20	60	42
		91	29
		136	39
		164	47
		378	32
Canwood	June 4	22	25
		79	58
		115	64
Ellerslie	June 21	37	36
		106	46
		348	37
Glendon	June 21	31	11
		108	24
		360	65

the same phenomena with elemental sulphur fertilized pot grown wheat while Janzen (1984) attributed lower recoveries of elemental sulphur fertilizers, in a potted canola experiment, to differential immobilization of the elemental sulphur. Since elemental sulphur fertilizers must pass "through" the soil biomass during oxidation, it is possible that a greater proportion of sulphur from this form is shunted into the organic sulphur pool than from sulphate sulphur forms (Janzen 1984).

From this experiment, it is apparent that each of the soils examined have significant capacity for elemental sulphur oxidation. It would also appear that the immobilization process is an important factor in field oxidation of elemental sulphur.

4.2.2 Effect of Time of Application on the Oxidation of Elemental Sulphur Sources

This experiment was undertaken to examine the effect of time of spring elemental sulphur application on its subsequent availability as sulphate.

Results from Breton (Table 13) and Ellerslie (Table 14) show that sulphur source recoveries are significantly different but the time of application was not significant. At both sites the treatment-application date interaction is significant indicating that the effect of time of application is dependent upon the sulphur material being added. The effect of application time was quite variable. At the Breton site (Table 13), the late application generally gave lower recoveries, at both sampling dates, except for the Bentonitic S (A) recovery in October where the late application

Table 13. Apparent net recovery (%), as sulphate-S, of incorporated fertilizer-S as affected by time of application at the Breton site in 1980.

		Breton Site SO_4-S kg ha^{-1} (% Net Recovery)			
		July 29, 1980		Oct. 17, 1980	
Treatment	Date of Application[a]	0-60 cm	Average	0-90 cm	Average
Control		25.9		34.6	
Fine S	May 2	34.0 (27)	32.0 (20)	45.2 (35)	44.6 (33)
	May 24	29.9 (13)		43.9 (31)	
Urea S (A)	May 2	39.0 (44)	36.9 (37)	59.6 (83)	54.8 (67)
	May 24	34.8 (30)		49.9 (51)	
Bentonitic S (A)	May 2	25.5 (0)	23.6 (-8)	35.1 (2)	39.7 (17)
	May 24	21.7 (-14)		44.2 (32)	
LSD[b]		1.3 (4)		2.2 (7)	
LSD[c]			1.8 (6)		1.4 (5)

[a] Application date is not significant.

[b] Treatment x application date interaction is significant.

[c] Treatment LSD at 5% level.

Table 14. Apparent net recovery (%), as sulphate-S, of incorporated fertilizer-S as affected by time of application at the Ellerslie site in 1980.

Treatment	Date of Application[a]	Ellerslie Site SO_4-S kg ha^{-1} (% Net Recovery)			
		July 28, 1980		Oct. 15, 1980	
		0–60 cm	Average	0–60 cm	Average
Control		21.6		15.9	
Fine S	May 2	39.6 (60)	42.1 (68)	48.6 (109)	44.3 (95)
	May 24	44.5 (76)		40.0 (80)	
Urea S (A)	May 2	29.9 (28)	33.8 (41)	44.2 (94)	47.3 (105)
	May 24	37.6 (53)		50.3 (115)	
Bentonitic S (A)	May 2	26.1 (15)	25.8 (14)	24.7 (29)	27.4 (38)
	May 24	25.4 (13)		30.0 (47)	
LSD[b]		1.7 (6)		7.8 (26)	
LSD[c]			2.1 (7)		4.2 (14)

[a] Application date is not significant.

[b] Treatment x application date interaction is significant.

[c] Treatment LSD at 5% level.

was higher. At the Ellerslie site (Table 14) the general trend has reversed with the majority of the treatments having lower recoveries from the early application except for the Fine S recovery in October where the early application recovery was significantly greater than the late application recovery.

The July results indicate that after 2 or 3 months of field "incubation", the average Fine S recovery was 20% at Breton and 68% at Ellerslie. Urea S (A) recovery was 37% at Breton and 41% at Ellerslie while Bentonitic S (A) recovery was zero and 14%. In October the Ellerslie site recoveries for Fine S and Urea S were roughly 100% and for Bentonitic S (A), 38%. At Breton in October, recoveries were 17% (Bentonitic S), 33% (Fine S) and 67% (Urea S (A)).

The recoveries from the spring applied finely divided elemental sulphur fertilizers were substantial and indicated that elemental sulphur could be an effective supplier of sulphate in the year of application.

4.2.3 Effect of Tillage on the Availability of Elemental Sulphur Sources

Two experiments were conducted to investigate the effects of field tillage on the rate of oxidation of some elemental sulphur sources.

4.2.3.1 Tillage Plot One

Both plots were set out in June of 1979 and the first sampling and tillage operations occurred on May 10, 1980 (Ellerslie) and June 29, 1980 (Canwood). A second sampling was completed on July 18, 1980 (Ellerslie) and August 14, 1980 (Canwood).

Results from Canwood (Table 15) and Ellerslie (Table 16) indicate that there are significant differences between sulphur source recoveries at both sampling dates. At the second sampling date, cultivation is significant for both experimental sites. For the first sampling date, recovery of sodium sulphate was 69% (Ellerslie) and 91% (Canwood). Fine S recovery was 28% (Ellerslie) and 68% at Canwood. Bentonitic S (A) recovery ranged from 10% at Canwood to 27% at Ellerslie. Thus, 11 to 12 months after application of the sulphur fertilizers, considerable quantities could be recovered as sulphate.

The August 14 sampling at Canwood was approximately six weeks after the plot was tilled. Excluding sodium sulphate, cultivation significantly increased the extractable sulphate of all the treatments including the Control. Sodium sulphate recovery was greater than 100% while Fine S ranged from 49% (untilled) to 66% (tilled). Bentonitic S also increased from 19% (untilled) to 29% (tilled). Thus, more than one year after application, significant quantities of fertilizer sulphur could be recovered and the amounts were further increased by cultivation.

The Ellerslie July 18 sampling was approximately eight weeks after the plot was tilled. Cultivation dramatically increased the extractable sulphate from all treatments including the control. Sodium sulphate recovery was 41% (untilled) to 106% (tilled). Fine S ranged from 27% (untilled) to 69% (tilled) while Bentonitic S (A) recovery was 9% (untilled) and 20% (tilled). Thus, more than one year after application, significant quantities of fertilizer sulphur

Table 15. Apparent net recovery (%), as sulphate-S, of incorporated fertilizer-S as affected by tillage; at the Canwood site in 1979. Tillage Experiment One.

Sulphur Source	Cultivation[a]	Canwood Site SO_4-S kg ha^{-1} (% Net Recovery) June 29, 1980 0-90 cm	Aug. 14, 1980 0-90 cm	Average
Control	No	7.2	16.7	18.6
	Yes		20.5	
Sodium sulphate	No	34.6 (91)	54.8 (127)	57.0 (128)
	Yes		59.1 (129)	
Fine S	No	27.8 (68)	31.3 (49)	35.9 (58)
	Yes		40.4 (66)	
Bentonitic S (A)	No	10.2 (10)	22.5 (19)	25.6 (23)
	Yes		29.1 (29)	
LSD[b]		4.9 (16)		6.2 (21)

[a] Cultivation is significant for Aug. 14 sampling. The cultivation x sulphur source interaction is not significant.

[b] Sulphur source LSD at 5% level for both dates.

Table 16. Apparent net recovery (%), as sulphate-S, of incorporated fertilizer-S as affected by tillage; at the Ellerslie site in 1979. Tillage Experiment One.

Sulphur Source	Cultivation[a]	Ellerslie Site SO_4-S kg ha^{-1} (% Net Recovery) May 10, 1980 0-60 cm	July 18, 1980 0-60 cm	Average
Control	No	18.9	7.3	13.1
	Yes		18.8	
Sodium sulphate	No	39.5 (69)	19.6 (41)	35.1 (73)
	Yes		50.5 (106)	
Fine S	No	27.2 (28)	15.3 (27)	27.5 (48)
	Yes		39.6 (69)	
Bentonitic S (A)	No	27.0 (27)	9.9 (9)	17.4 (14)
	Yes		24.9 (20)	
LSD[b]		0.8 (3)		1.0 (3)
LSD[c]			1.8 (6)	

[a] Cultivation is significant for July 18 sampling.

[b] Sulphur source LSD at 5% level for both dates.

[c] Sulphur source x cultivation interaction is significant.

could be recovered and the amounts were dramatically increased by cultivation.

These results indicate tillage caused a significant increase in recoverable sulphate probably through increased oxidation and mineralization.

4.2.3.2 Tillage Plot Two

Fallow field plots received sulphur fertilizer in October 1980 and two tillage operations were completed before the soil became frozen. The plots were sampled in the early spring (May) and fall (September or October) of 1981.

Results for Breton (Table 17), Canwood (Table 18) and Glendon (Table 19) are variable for no apparent reason. Cultivation was not significant for either sampling date at all three plot sites. Sulphur sources were significantly different at each sampling date for each site except for the second sampling (October 31) at Breton where the cultivation-sulphur source interaction was significant.

The first sampling date allows an evaluation of overwinter oxidation of elemental sulphur. Recovery of Fine S was from 7 to 10% while recovery of Urea S (B) was from less than 0 to 16%. It would appear that while overwinter oxidation occurs, it is minimal. At Breton, sodium sulphate recovery was a very low 34% indicating losses due to leaching. Recovery at Canwood was 61% while Glendon recovery was 134%.

Fall recovery of sulphur at the Breton site was minimal presumably because there were leaching losses due to heavy spring and

Table 17. Apparent net recovery (%), as sulphate-S, of incorporated fertilizer-S as affected by tillage, at the Breton site in 1981. Tillage Experiment Two.

Sulphur Source	Cultivation[a]	Breton Site SO_4-S kg ha^{-1} (% Net Recovery)			
		May 2, 1981		Oct. 31, 1981	
		0-30 cm	Average	0-60 cm	Average
Control	No	9.3	9.5	17.8	18.3
	Yes	9.7		18.8	
Sodium sulphate	No	20.6 (38)	19.8 (34)	24.8 (23)	21.6 (11)
	Yes	18.9 (31)		18.3 (-2)	
Fine S	No	11.1 (6)	11.5 (7)	22.2 (15)	22.6 (14)
	Yes	11.9 (7)		22.9 (14)	
Urea S (B)	No	14.3 (17)	14.4 (16)	26.1 (28)	22.3 (13)
	Yes	14.4 (16)		18.5 (-1)	
LSD			4.0 (13)[b]	5.2 (17)[c]	

[a] Cultivation is not significant at either sampling date.

[b] Sulphur source LSD at 5% level.

[c] Sulphur source x cultivation interaction at 5% level. Sulphur source is not significant.

Table 18. Apparent net recovery (%), as sulphate-S, of incorporated fertilizer-S as affected by tillage, at the Canwood site in 1981. Tillage Experiment Two.

Sulphur Source	Cultivation[a]	Canwood Site SO_4-S kg ha^{-1} (% Net Recovery)			
		May 10, 1981		Sept. 26, 1981	
		0-30 cm	Average	0-60 cm	Average
Control	No	7.5	6.4	3.8	4.1
	Yes	5.3		4.4	
Sodium sulphate	No	24.9 (58)	24.6 (61)	21.7 (60)	25.1 (70)
	Yes	24.2 (63)		28.5 (80)	
Fine S	No	10.4 (10)	9.5 (10)	27.0 (77)	25.5 (71)
	Yes	8.5 (11)		24.0 (65)	
Urea S (B)	No	7.7 (1)	8.3 (6)	11.9 (27)	12.7 (29)
	Yes	8.9 (12)		13.5 (30)	
LSD[b]			4.5 (15)		9.0 (30)

[a] Cultivation is not significant at either date.

[b] Sulphur source LSD at 5% level.

Table 19. Apparent net recovery (%), as sulphate-S, of incorporated fertilizer-S as affected by tillage, at the Glendon site in 1981. Tillage Experiment Two.

Sulphur Source	Cultivation[a]	Glendon Site SO_4-S kg ha^{-1} (% Net Recovery)			
		May 1, 1981		Oct. 6, 1981	
		0-30 cm	Average	0-60 cm	Average
Control	No	10.4	7.9	11.4	12.4
	Yes	5.4		13.4	
Sodium sulphate	No	45.3 (116)	48.0 (134)	42.2 (103)	44.5 (107)
	Yes	50.7 (151)		46.8 (111)	
Fine S	No	13.8 (8)	11.0 (10)	30.7 (64)	27.2 (49)
	Yes	9.2 (13)		23.6 (34)	
Urea S (B)	No	5.9 (-15)	6.3 (-6)	10.9 (-2)	13.2 (3)
	Yes	6.6 (4)		15.4 (7)	
	LSD[b]		4.6 (15)		5.2 (17)

[a] Cultivation is not significant at either sampling date.

[b] Sulphur source LSD at 5% level.

summer rains. Fall recovery of sodium sulphate was 70% at Canwood and 107% at Glendon. Fine S recoveries for Canwood and Glendon were 71 and 49%, respectively, while Urea S recovery was 3 and 29%, respectively.

The significant quantities of Fine S recovered in the fall indicate that conditions over the summer were conducive to elemental sulphur oxidation at the Canwood and Glendon sites.

4.2.4 Discussion

The results from the several fallow field experiments are highly variable and this should be expected. Because of the dynamic nature of sulphur and the fact that elemental sulphur is biochemically oxidized, any fallow field evaluation of elemental sulphur oxidation is fraught with the interfering processes of immobilization, mineralization and leaching. The elemental sulphur material applied and the method of application further complicate the situation. In spite of these difficulties, elemental sulphur fertilizers comprised of fine particles mixed throughout the soil in the spring will be 30 to 100% oxidized by the fall. As previously discussed in section 4.1.1.4, elemental sulphur products that leave aggregations of fine particles (Urea S, Bentonitic S) will be more slowly oxidized than those that can be mixed throughout the soil (Fine S).

Elemental sulphur mixed into the soil in the fall gave little recovery, as sulphate, the following spring (at the time of seeding) but was significantly oxidized by the next fall (Tillage Plot Two).

Occasionally cultivation increased the amount of elemental sulphur oxidized and as often it did not. Occasionally rather large amounts of elemental sulphur and sulphate sulphur can be immobilized (discussed in section 4.2.1) and subsequently mineralized as a result of cultivation (Tillage Plot One).

The results from these experiments generally have oxidation rates comparable to similar experiments reported in the literature for other parts of the Prairie Provinces (Ukrainetz 1982, Janzen et al. 1982). In general, the soils tested in these experiments have significant oxidative capacity.

4.3 CROPPED FIELD EXPERIMENTS

4.3.1 Effect of Time of Application of Elemental Sulphur on Its Availability to Barley

A barley experiment was established on a moderately deficient soil at the Glendon site, in the spring of 1981, to evaluate the effect of time of spring elemental sulphur application on its subsequent availability as plant available sulphate.

Urea S (B) supplied sulphur at 11.2 kg ha^{-1} or 70 kg ha^{-1} on May 1 (early) and May 23 (late). Sodium sulphate was used as the sulphate control and Urea supplied nitrogen at 112 kg ha^{-1} to all treatments excluding the control.

Barley yield was significantly increased by late urea application but not by the early applied urea (Table 20; Appendix I) while sulphur uptake was increased by 1.0 kg ha^{-1} and 1.2 kg ha^{-1}, respectively. Despite the increased utilization of soil sulphur, the

Table 20. 1981 and 1982 yields, S uptakes and N:S ratios of barley with annual spring application of nitrogen (112 kg N ha^{-1}) in 1981 and 1982, and spring 1981 application of sulphur (11.2 or 70 kg ha^{-1}) at the Glendon site.

Treatment	Application of S		1981			1982		
	Time Added in 1981	Amount (kg ha^{-1})	Grain Yield (tonne ha^{-1})	Total S Uptake (kg ha^{-1})	N:S Ratio	Grain Yield (tonne ha^{-1})	Total S Uptake (kg ha^{-1})	N:S Ratio
Control		0	1.82	3.8	10.1	1.56	4.2	8.8
Urea	May 1	0	2.29	4.8	16.0			
Urea	May 23	0	2.48	5.0	17.6	2.99	8.0	11.6
Urea + sodium sulphate	May 1	11.2	3.15	9.1	10.0	3.36	9.6	10.4
Urea + sodium sulphate	May 23	11.2	2.95	9.3	10.9	3.45	9.2	11.2
Urea + Urea S	May 1	11.2	2.75	5.5	14.4	3.67	9.8	11.0
Urea + Urea S	May 23	11.2	2.68	5.3	15.6	3.43	10.2	10.8
Urea S (B)	May 1	70	2.96	8.9	10.3	3.48	12.4	8.0
Urea S (B)	May 23	70	2.96	5.9	16.1	3.41	12.9	7.0
		LSD	0.49	1.2	2.9	0.54	1.8	1.1

N:S ratios widened to 16.0 (early) and 17.6 (late). Early and late application of urea plus sodium sulphate gave significant yield increases, significantly greater total sulphur uptake and N:S ratios of 10.0 and 10.9.

Comparing the sulphur uptake of the crop in the plot treated with early urea and in the plot treated with early urea plus sodium sulphate, the sulphate recovery from sodium sulphate was 38% (late recovery was also 38%). For early Urea plus Urea S (B) a sulphur recovery was 5% while late recovery was 3%. Crop sulphur uptake (8.9 kg ha^{-1}) from early Urea S applied alone (S at 70 kg ha^{-1}) was similar to the uptake from sodium sulphate (9.1 kg ha^{-1}) as was the N:S ratio (10:3) and sulphur recovery from the S application of 70 kg ha^{-1} application was 6%. The late Urea S (alone) treatment did not significantly increase sulphur uptake (5.9 kg) or decrease N:S ratio (16:1).

4.3.2 Residual Availability of Antecedent Applications of Elemental Sulphur to Barley

The Glendon barley plot was maintained through the 1982 growing season to investigate the residual availability of elemental sulphur to barley.

Urea (supplying N at 112 kg ha^{-1}) was applied in 1982 to each subplot, excluding the control, with no additional sulphur fertilizer added. This allowed for an evaluation of the residual characteristics of two rates of 1981 applied elemental sulphur (11.2 and 70 kg ha^{-1}) and one rate (11.2 kg ha^{-1}) of sodium sulphate.

Barley grain yield was significantly increased (1.43 tonne ha^{-1}) by urea application (Table 20; Appendix II) and sulphur uptake was significantly increased by 3.8 kg ha^{-1} while the N:S ratio increased to 11.6. All of the 1981 applications of Urea S gave equivalent or significantly greater sulphur uptake when compared to sodium sulphate. Sodium sulphate recovery in 1982 was 11 to 14% giving two-year totals of 49 and 52% for the early and late 1981 treatments. Recoveries of sulphate from the early and late 11.2 kg S ha^{-1} applications of Urea S were 16 and 20%, respectively, for two-year totals of 21 and 23%. Total crop uptake of sulphur was greatest from the 1981 Urea S treatments that supplied sulphur at 70 kg ha^{-1} and the crop N:S ratios (7.0 and 8.0) reflected the presence of sufficient crop available sulphur. Total two-year recoveries were 12 and 8% for early and late 1981 sulphur applications of Urea S (70 kg ha^{-1}).

4.3.3 Availability of Surface Applied Elemental Sulphur to Bromegrass

Field plots were established in the spring of 1981 on a native bromegrass (Bromus inermis) stand to evaluate the short- and long-term availability of surface applied elemental sulphur (11.2 kg ha^{-1}). Fertilization of the plots continued in the spring of 1982 and 1983 (refer to section 3.5.3 for details).

In 1981 (Table 21, Appendix III and IV) the sulphur recovery from the Urea plus sodium sulphate treatment was 4.9 kg ha^{-1} (44%). This value was calculated by deducting the sulphur uptake in bromegrass receiving urea alone from the sulphur uptake in bromegrass on

Table 21. 1981, 1982 and 1983 yields, S uptakes and N:S ratios of bromegrass with annual spring application of nitrogen (112 kg ha^{-1}) and sulphur (11.2 kg ha^{-1}) at the Canwood site.

Treatment	Hay 1981			Hay 1982			Hay 1983*		
	Yield (tonne ha^{-1})	S Uptake (kg ha^{-1})	N:S Ratio	Yield (tonne ha^{-1})	S Uptake (kg ha^{-1})	N:S Ratio	Yield (tonne ha^{-1})	S Uptake (kg ha^{-1})	N:S Ratio
Control	1.71	2.1	11.6	2.34	2.8	9.9	1.16	1.1	16.0
Urea	1.08	1.7	32.9	3.13	2.8	20.4	0.86	0.5	39.5
Sodium sulphate	1.79	2.9	8.3	2.60	4.4	7.7	1.17	2.1	7.8
Urea and sodium sulphate	4.28	6.6	13.3	5.26	7.9	10.2	2.58	6.1	6.9
Urea and Urea S	3.14	2.9	25.4	5.41	7.0	11.6	3.06	4.4	10.0
Urea and Fine S	3.17	2.6	29.6	5.45	6.5	13.7	2.50	2.5	13.7
LSD	0.71	1.2	4.8	0.69	0.9	4.3	0.98	1.6	3.2

* 1981 and 1982 values are totals of two cuts, one in mid-summer (late July - early August) and the second in mid-fall (late September). The 1983 values are from a single cut taken in early August.

the plot that received urea plus sodium sulphate. Recovery of sulphur from Fine S and Urea S was 1.2 (11%) and 0.9 (8%) kg ha^{-1}, respectively. Urea and sodium sulphate yield (4.3 tonne ha^{-1} was significantly greater than Urea S 3.1 tonne ha^{-1}) and Fine S (3.2 tonne ha^{-1}). Similarily, the N:S ratios of bromegrass from the Fine S (29.6) and Urea S (25.4) treatments were significantly higher than the Urea plus sodium sulphate treatment (13.3).

Percent recovery values for 1982 and 1983 could not be calculated since fertilizers were added each spring. Yields of bromegrass in 1982 and 1983 were not significantly different for the three treatments receiving urea and sulphur fertilizer. The 1982 Urea plus sodium sulphate and Urea S treatments had sulphur uptakes of 5.1 and 4.2 kg S ha^{-1}, respectively, that were significantly greater than the other treatments. N:S ratios of the nitrogen plus sulphur treatments were not significantly different. In 1983 the Urea plus sodium sulphate treatment sulphur uptake was 5.6 kg ha^{-1} which was significantly greater than Urea S uptake (3.9 kg ha^{-1}) and the 2.0 kg ha^{-1} sulphur uptake of the Fine S treatment. The 1983 N:S ratio of Fine S (13.7) was significantly greater than Urea S (10.0) and Urea plus sodium sulphate (6.9).

4.3.4 Discussion

Previous to the 1981 growing season three western Canadian studies (Nyborg et al. 1980, Noellemeyer et al. 1981, Swan and Soper 1981) indicated that there was minimal short-term release of sulphate from elemental sulphur products. Toogood et al. (1962) observed that

the average long-term leguminous hay yields (1940-1961) from plots fertilized every second year with flowers of elemental sulphur (15.7 kg ha^{-1}) was equal to or greater than yields from plots receiving gypsum or sodium sulphate. Additionally, the long-term average yields of cereal grains grown after hay on the elemental sulphur plots were equal to or greater than the average yields obtained from plots treated with gypsum and sodium sulphate. Several subsequent papers, from the United States, indicated a significant residual component associated with elemental sulphur applications (Fox et al. 1964, Jones and Ruckmam 1966, 1969, Kiemnec et al. 1981).

The Glendon barley plot (1981 and 1982) and the Canwood Bromegrass plot (1981, 1982 and 1983) both display the delayed availability and residual characteristics of elemental sulphur. With the barley plot in 1981 (Table 20, Appendix I) only barley grown on subplots receiving early applications of Urea S (sulphur supplied at 70 kg ha^{-1} utilized similar amounts of sulphur as Urea plus sodium sulphate. This was reflected in the total S uptake values and N:S ratios. Interestingly, the same treatment applied three weeks later (at the time of seeding) did not significantly increase the total S uptake (over the late Urea only treatment) or reduce the N:S ratio even though sulphur was applied at 70 kg ha^{-1}. This emphasizes the delay of elemental sulphur availability.

In 1982 all of the residual Urea S treatments had total sulphur uptake values equal to or significantly greater than the residual Urea plus sodium sulphate treatments (Table 20, Appendix II). The N:S ratios were also the same (10.4-11.2) or significantly

less (7.0-8.0). The Urea S alone (supplying S at 70 kg ha^{-1}, 1981) treatments had significantly greater total S uptake values and significantly reduced N:S ratios than the sodium sulphate and 11.2 kg ha^{-1} rate of Urea S thereby indicating sufficient sulphur availability (Stewart 1969).

The bromegrass plots exhibited similar results with elemental sulphur fertilizers applied to the surface of a severely deficient soil (Table 21, Appendix III and IV). In 1981 the Urea plus elemental sulphur fertilizers (11.2 kg ha^{-1}) gave significantly lower yields, lower total sulphur uptake values and wider N:S ratios when compared to Urea plus sodium sulphate. This further emphasizes the delay in availability of applied elemental sulphur.

In 1982 the yields and N:S ratios of the three Urea plus sulphur treatments were not significantly different. Total sulphur uptake from the Fine S treatment was significantly lower than the sulphur uptake values for the Urea plus sodium sulphate and Urea plus Urea S treatments. The 1982 data indicate a strong residual effect of the previous year's application of Fine S or an increased oxidative capacity of the Canwood Brome soil.

The 1983 yield data appear to confuse the situation but when certain environmental and procedural factors are taken into account, the picture is less cloudy. Only one cut was taken in early August (1981 and 1982 values are totals of two cuts), so the data does not reflect the effects of a full growing season on oxidation of the applied elemental sulphur. The spring of 1983 was exceedingly dry followed by heavy rains in late June and early July. These factors

would minimize early spring oxidation of elemental sulphur (due to dryness) and with the heavy rains the rapidly growing bromegrass would require amounts of sulphur that the elemental sulphur fertilizers did not seem able to supply. Yields from the nitrogen plus sulphur treatments were not significantly different but sulphur uptake from the elemental sulphur fertilizers was significantly lower and the N:S ratios wider when compared to nitrogen plus sodium sulphate.

The elemental sulphur products used in these experiments included Urea S (B), Urea S (C) and Fine S (see Table 3). Discussion in section 4.1.1.4 indicated that Urea S leaves "aggregations" of fine elemental sulphur particles in the soil. Before these particles can be fully exposed to extensive microbial oxidation, the "aggregations" must be fractured so that the particles can be mixed throughout the soil. The poor utilization of Urea S in the first year of barley at the Glendon site can be attributed to poor dispersion of the fine elemental sulphur particles contained within Urea S. The 1982 barley plot showed that after one year there was a significant residual effect of the Urea S (especially the heavy rate). Increased time for oxidation and further disruption of the sulphur aggregations through tillage are important aspects of this residual effect.

Other factors impede the rate of oxidation of surface applied elemental sulphur. Urea S and Fine S applied to the grass-soil surface have reduced surface contact with the soil and the microbes within. Conditions at the surface fluctuate even more

widely than they do within the soil and are less favorable to oxidation. Despite these limitations to elemental sulphur oxidation, the two materials can supply sufficient quantities of sulphur to the growing hay crops. Fine S often resulted in lower recoveries of total sulphur uptake and wider N:S ratios. This could be attributed to its larger particle size as compared to the individual particles in Urea S.

A positive attribute of surface application of Urea S would appear to be the effect of rain on the dispersion of the elemental sulphur aggregates that remain after Urea dissolution. Rain drops carry adequate energy and are sufficiently repetitive to effectively disperse the Urea S aggregates and carry the fine particles to the soil surface. For short-term response from elemental sulphur fertilizers that leave aggregations of fine particles (Urea S, Bentonitic S), the best method of application may be to surface broadcast and leave the sulphur material on the soil surface where the elements of nature can disperse the particles prior to incorporation.

5. DISCUSSION

This study was undertaken in order to evaluate the environmental, microbiological and cultural factors that affect the oxidation and subsequent availability of elemental sulphur to growing plants.

Elemental sulphur must be biologically oxidized to sulphate before it can be utilized by crops. Thus, oxidation is highly dependent upon the soil environment, principally temperature and moisture content. Incubation studies were undertaken to evaluate temperature and moisture effects on elemental sulphur oxidation.

A parabolic relationship between soil moisture and sulphur oxidation was observed with maximum rates of oxidation occurring near soil field capacity. It was also noted that significant quantities of elemental sulphur were oxidized at all of the experimental conditions of 40% F.C., 65% F.C., 90% F.C. and 120% F.C. at 24°C. Over the growing season, the moisture content of Prairie Province soils generally falls within the lower portion of this range.

As temperature increased from 6°C to 36°C, oxidation rate increased so that the 36°C treatment oxidized 4.8 to 6.0 times the amount oxidized at 6°C. The actual increase due to temperature was probably much higher. As Janzen (1984) points out, methods that measure mass of elemental sulphur oxidized over time bias results toward the less optimum conditions since the amount of elemental sulphur remaining after a sampling date will always be lower in conditions that favour higher oxidation rates. Thus, for the next

sampling period, less elemental sulphur is available for microbial oxidation in the containers incubated under optimal conditions as compared to the elemental sulphur remaining under the less favorable conditions. Nevertheless, this experiment demonstrates the important role temperature plays in elemental sulphur oxidation. With decreasing temperature, the Breton soil exhibited a progressively slowed restoration of oxidative capacity which further illustrated the importance of soil temperature on elemental sulphur oxidation. An overwinter field "incubation" resulted in very low recoveries of elemental sulphur early the following spring, presumably because the soil temperatures were too low for substantial oxidation. The same experiment (and others) had substantial recovery of sulphate in the subsequent fall after a much warmer summer field "incubation".

Incubation experiments illustrated the effect of elemental sulphur placement on oxidation. Any method that restricts the exposed surface area of the elemental sulphur reduced oxidation rate because biological oxidation requires direct contact between microbial cells and sulphur particles (Vogler and Umbreit 1941).

Therefore, any fertilizer material that left small aggregations of finely divided elemental sulphur particles in the soil also reduced the surface area exposed to microbial oxidation and reduced oxidation rates. This fact was highlighted by greatly increased sulphate recoveries from placed Fine S or Urea S treatments that had been thoroughly "cultivated". In the field, Urea S often, and Bentonitic S nearly always, had lower recoveries of sulphate when compared to broadcast Fine S. Often field cultivation, and a less

intense incubation experiment "cultivation", did not disrupt the sulphur aggregations sufficiently to increase oxidation. Field cultivation occasionally increased oxidation of Fine S but often did not. Cultivation seemed to have a greater effect on mineralization and immobilization.

Laboratory incubations of long duration (up to 12 weeks) and field experiments were often confounded by pronounced immobilization-mineralization effects. Incubations of shorter duration (30 days) seemed to have minimal interference from immobilization and mineralization especially at the earlier sampling dates. Fallow field experiments were further confounded by leaching losses of sulphate generated by elemental sulphur oxidation.

Elemental sulphur fertilizer applied to barley and bromegrass plots displayed a delay in availability to the growing crops as well as a significant residual quality. In the first year of a two-year barley experiment, an application, three weeks prior to seeding, of Urea S (supplying S at 70 kg ha^{-1}) was the only elemental sulphur treatment that allowed barley sulphur uptake equal to that from an application of sodium sulphate (supplying S at 11.2 kg ha^{-1}). In the following crop year all of the Urea S treatments supplied equal or greater amounts, as compared to sodium sulphate, of residual sulphur.

Results for a three-year bromegrass plot receiving annual surface applications of Urea nitrogen at 112 ha^{-1} and sulphur at 11.2 kg ha^{-1} (supplied as sodium sulphate, Fine S or Urea S) indicated that the elemental sulphur fertilizers had a significant

residual quality because yields in the second and third years were equal to the nitrogen plus sodium sulphate treatment yields.

An incubation experiment utilizing soils that had previously (1 to 2 years) received agronomic rates of elemental sulphur and soils that had never received elemental sulphur, indicated that previously treated soils oxidize subsequent applications of elemental sulphur more quickly in the laboratory. This finding leads one to speculate that, in the field, elemental sulphur applied to previously exposed soils may be more quickly oxidized than the initial field application.

A method of fertilizer application was formulated for short-term response to elemental sulphur fertilizers that leave "aggregations" of fine sulphur particles. The suggested method involves leaving the surface broadcast sulphur on the soil surface so that the elements of nature can disperse the "aggregation" particles prior to incorporation.

6. CONCLUSIONS

The results obtained from the laboratory incubation experiments and the field experiments allow the following conclusions:

1) For short-term elemental sulphur fertilizer availability only those products containing very finely divided particles will supply sufficient sulphate and then only if thoroughly mixed throughout the soil. In severely deficient soils, applications three to four times the recommended amount of sulphate sulphur fertilizer will be required. Alternatively, a split application of sulphate and elemental sulphur would be effective.
2) Any method of application, or elemental sulphur material, that reduces the exposed surface area of the applied elemental sulphur will reduce the rate of oxidation.
3) Elemental sulphur has a significant residual availability to crops grown subsequent to the year of application.
4) Previously applied agronomic levels of elemental sulphur seems to increase the rate of oxidation of subsequent additions of elemental sulphur.
5) Soil temperatures and moisture contents that allow crop growth will encourage oxidation of elemental sulphur.
6) All of the soils evaluated in this study oxidized significant amounts of finely divided sulphur.
7) Cultivation of soils treated with elemental sulphur occasionally increases the apparent rate of oxidation but often it does not.

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Appendix I. 1981 yields, S uptakes and N:S ratios of barley with different times of application of N (112 kg ha^{-1}) and S (11.2 kg or 70 kg ha^{-1}) at the Glendon site.

Treatment	Time	Yield (tonne ha^{-1})			S Uptake (kg ha^{-1})			N:S Ratios		
		Straw	Grain	Total	Straw	Grain	Total	Straw	Grain	Total
Nil		1.44	1.82	3.25	1.3	2.5	3.8	5.8	12.3	10.1
Urea	May 1	2.05	2.29	4.34	1.4	3.4	4.8	13.9	15.8	16.0
Urea	May 23	2.23	2.48	4.71	1.4	3.6	5.0	16.0	18.2	17.6
Urea + sodium sulphate	May 1	2.68	3.15	5.83	3.7	5.4	9.1	5.5	13.1	10.0
Urea + sodium sulphate	May 23	2.61	2.95	5.56	3.7	5.6	9.3	7.1	12.9	10.9
Urea + Urea S (B)	May 1	2.17	2.75	4.91	1.3	4.2	5.5	12.2	15.0	14.4
Urea + Urea S (B)	May 23	2.01	2.68	4.69	1.3	4.0	5.3	12.8	16.6	15.6
Urea S (B)	May 1	2.42	2.96	5.37	3.4	5.5	8.9	6.1	12.8	10.3
Urea S (B)	May 23	2.25	2.96	5.21	1.4	4.5	5.9	12.5	17.2	16.1
LSD		0.38	0.49	0.81	0.4	0.9	1.2	3.9	2.9	2.9

Appendix II. 1982 yields, S uptakes and N:S ratios of barley with 1981 antecedent S application (11.2 kg or 70 kg ha^{-1}) and 1982 applied N (112 kg ha^{-1}) at the Glendon site.

Treatment	Application of S		Yield (tonne ha^{-1})			S Uptake (kg ha^{-1})			N:S Ratios		
	Time Added in 1981	Amount (kg ha^{-1})	Straw	Grain	Total	Straw	Grain	Total	Straw	Grain	Total
Nil			1.40	1.56	2.97	1.9	2.3	4.2	5.8	11.3	8.8
Urea	May 23	0.0	2.82	2.99	5.81	2.9	5.1	8.0	10.0	12.7	11.6
Urea + sodium sulphate	May 1	11.2	3.09	3.36	6.45	3.6	5.9	9.6	8.3	11.7	10.4
Urea + sodium sulphate	May 23	11.2	2.87	3.45	6.32	3.3	5.9	9.2	9.1	12.4	11.2
Urea + Urea S (B)	May 1	11.2	3.40	3.67	7.07	3.6	6.1	9.8	9.0	12.1	11.0
Urea + Urea S (B)	May 23	11.2	3.32	3.43	6.75	4.2	6.0	10.2	8.2	12.5	10.8
Urea S (B)	May 1	70.0	2.98	3.48	6.46	6.2	6.3	12.4	4.3	11.7	8.0
Urea S (B)	May 23	70.0	3.34	3.41	6.75	6.4	6.5	12.9	4.0	10.1	7.0
LSD			0.61	0.54	0.98	1.4	0.9	1.8	1.9	1.0	1.1

Appendix III. 1981 yields, S uptakes and N:S ratios of bromegrass with annual spring application of nitrogen (112 kg ha^{-1}) and sulphur (11.2 kg ha^{-1}) at the Canwood site.

Treatment	Cut One 1981			Cut Two 1981			Total 1981		
	Yield (tonne ha^{-1})	S Uptake (kg ha^{-1})	N:S Ratio	Yield (tonne ha^{-1})	S Uptake (kg ha^{-1})	N:S Ratio	Yield (tonne ha^{-1})	S Uptake (kg ha^{-1})	N:S Ratio
Control	1.1	1.5	11.8	0.58	0.6	10.8	1.71	2.1	11.6
Urea	1.57	1.2	38.1	0.51	0.4	20.8	2.08	1.7	32.9
Sodium sulphate	1.14	2.0	8.3	0.65	0.9	7.5	1.79	2.9	8.3
Urea and sodium sulphate	3.29	5.5	14.3	1.00	1.2	10.6	4.28	6.6	13.3
Urea and Urea S	2.14	1.9	31.2	1.00	1.0	14.2	3.14	2.9	25.4
Urea and Fine S	2.28	1.8	36.4	0.89	0.8	14.7	3.17	2.6	29.6
LSD	0.66	1.2	7.6	0.20	0.2	2.5	0.71	1.2	4.8

Appendix IV. 1982 yields, S uptakes and N:S ratios of bromegrass with annual spring application of nitrogen (112 kg ha^{-1}) and sulphur (11.2 kg ha^{-1}) at the Canwood site.

Treatment	Cut One 1982			Cut Two 1982			Total 1982		
	Yield (tonne ha^{-1})	S Uptake (kg ha^{-1})	N:S Ratio	Yield (tonne ha^{-1})	S Uptake (kg ha^{-1})	N:S Ratio	Yield (tonne ha^{-1})	S Uptake (kg ha^{-1})	N:S Ratio
Control	0.89	1.2	11.9	1.45	1.6	8.4	2.34	2.8	9.9
Urea	1.43	1.1	34.8	1.70	1.8	16.0	3.13	2.8	20.4
Sodium sulphate	1.03	2.1	8.6	1.57	2.3	6.7	2.60	4.4	7.7
Urea and sodium sulphate	3.62	5.8	10.8	1.89	2.1	9.2	5.26	7.9	10.2
Urea and Urea S	3.62	4.9	13.2	1.79	2.1	7.9	5.41	7.0	11.6
Urea and Fine S	3.43	4.5	15.3	2.02	2.0	10.2	5.45	6.5	13.7
LSD	0.69	1.1	8.6	0.37	0.5	2.7	0.98	1.6	3.2

Appendix V. Fractionation of fine sublimed elemental sulphur by mechanical sieving.

Fraction Particle Size[1]	% Fine S Collected		
	Rep. One	Rep. Two	Average
>250 um	0.0	0.0	0.0
250–150 um	21.7	24.1	22.9
150– 75 um	56.2	60.1	58.2
75– 53 um	7.5	7.8	7.7
53– 45 um	8.8	5.5	7.2
< 45 um	5.8	2.5	4.2

[1] Particle size fraction designations represent the smallest particle diameter to be collected on the sieve.

Appendix VI. Method for fractionation of Bentonitic S sources.

Duplicate 250 g samples of Bentonitic S (B) and Bentonitic S (C) were weighed and placed in beakers, covered with water and left standing to dry, at room temperature, for 24 hours. Sufficient water was added to cover the sulphur material with approximately 2 cm of water and the beakers were then placed in an oven at 38°C to dry. The sulphur materials were subsequently wetted and dried four additional times over a four-day period. Each sample was then mechanically sieved and the particle size fractions calculated.

Fractionation of Bentonitic S (B) and Bentonitic S (C) by mechanical sieving:

Fraction Particle Size[1]	% Bentonitic S Collected					
	Bentonitic S (B)			Bentonitic S (C)[2]		
	Rep. 1	Rep. 2	Ave.	Rep. 1	Rep. 2	Ave.
> 2,000 um	0	0	0	26	24	25
2,000–1,180 um	19	15	17	13	17	15
1,180–600 um	35	37	36	14	15	15
600–250 um	19	20	20	18	14	16
250–150 um	12	13	13	13	12	13
150–75 um	10	12	11	11	12	12
75–45 um	3	2	3	4	4	4
< 45 um	1	1	1	1	1	1

1 Particle size fraction designations represent the smallest particle diameter to be collected on the sieve.

2 Bentonitic S (C) is similar to Bentonitic S (A) which is no longer commercially available.